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The effect of thermal-mechanical processing of the exfoliation corrosion resistance of an aluminum-zinc-magnesium-copper alloy

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THE EFFECT OF THERMAL-MECHANICAL PROCESSING ON THE EXFOLIATION CORROSION RESISTANCE OF AN ALUMINUM-ZINC-MAGNESIUM-COPPER ALLOY

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ABSTRACT

The objective of this investigation was to evaluate the effect of thermal-mechanical processing on the exfoliation corrosion resistance of an aluminum-zinc-magnesium-copper alloy. The aluminum alloy used was commercially available 7178 alloy in two thicknesses, 0.390 inch thick and 0.250 inch thick. This material was reduced 10, 25 and 40% in thickness in both the annealed and solution heat treated conditions. The material was then aged to a high strength condition, T6, or T8, in the case of the material reduced in the solution heat treated condition. The effect of the thermal-mechanical processing on the exfoliation corrosion resistance was then evaluated by exposing 3 by 6 inch specimens to both an intermittent acidified salt spray test and a salt spray plus sulfur dioxide gas test.

Increasing amounts of mechanical deformation of material in the solution heat treated condition resulted in progressively lower resistance to exfoliation corrosion. As the amount of deformation of the annealed material increased to a point where recrystallization resulted during the subsequent solution heat treatment, the exfoliation corrosion resistance improved.

The aging schedules developed for the T8 condition resulted in strengths equivalent to the high strength T6 condition without significant losses in ductility.

It was also demonstrated that the salt spray plus sulfur dioxide test was a considerably more severe exfoliation test environment than the intermittent acidified salt spray test.

PREFACE

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I. INTRODUCTION

A. Statement of Problem

Commercial and military aircraft make liberal use of high strength aluminum alloys in structural applications. One such high strength alloy, aluminum-zinc-magnesium-copper (7178), is used extensively on exterior skins, primarily wing skins. This alloy in the high strength condition (T6) has been reported to be susceptible to exfoliation corrosion (1,2,3,4), primarily around countersunk fastener holes. Exfoliation corrosion manifests itself in blistering and delamination of the metal surrounding fastener holes, which results in degradation of the structural integrity of the part and consequently expensive replacement or repair of such parts. Special aging procedures have been developed (5,6,7,8) to improve the resistance of alloys found susceptible to exfoliation corrosion. However, these special aging treatments result in a reduction in the strength of the alloy which imposes weight penalties when using the special condition in designing aircraft parts.

B. Reason For Selection

To design weight efficient aircraft structure using the high strength aluminum alloy (7178), a need exists to improve the exfoliation corrosion resistance of this alloy without impairing the mechanical properties. The exfoliation corrosion resistance can be improved by any of three techniques: (a) by changing the fabrication techniques which affect the microstructure of the alloy to one which is not susceptible to exfoliation; (b) excluding the corroding environment by means of coatings or protective treatments; (c) or by means of special aging treatments which result in lower mechanical properties.

This study will attempt to improve the exfoliation corrosion resistance of the 7178 aluminum alloy, without reducing the strength, by developing a T8 condition that results in a microstructure which is not susceptible to exfoliation corrosion. The T8 condition is achieved by cold working the material after solution heat treatment and water quenching but prior to aging.

Previous investigators (9) have found this approach to be successful in improving the fatigue strength and stress corrosion resistance of the similar 7075 aluminum alloy.

Susceptibility to exfoliation corrosion will be evaluated by exposure to an acidified salt spray and a more severe test which consists of a continuous spray of 5% sodium chloride with periodic additions of sulfur dioxide gas.

The aging cycle will be monitored using Rockwell hardness testing and electrical conductivity tests.

II. HISTORICAL BACKGROUND

A. Exfoliation Corrosion

1. Reported Incidences

Exfoliation has been observed in many aluminum alloy systems. One of the first documentations of exfoliation corrosion in this country was by Reinhart (2) in 1954. Reinhart reported intergranular attack accompanied by exfoliation in commercial and military aircraft wing skins fabricated from alclad and bare 75S-T6 and alclad 24S-T3. The attack generally originated around fastener holes in areas of the wing swept by exhaust gases from the engines. Lead bromide was among the products of combustion and was suspected to be the chief cause of this corrosion. Other reports of exfoliation damage to aircraft structures (1,4) followed the same pattern, i.e., intergranular corrosion accompanied by internal corrosion products which forcibly expanded unaffected surface areas. The attack was always associated with fastener holes, and followed a laminar pattern along grain boundries thereby degrading the structural integrity of the metal. Exfoliation attack in less advanced stages showed as blisters below painted surfaces. Early attempts to repair or inhibit exfoliation corrosion proved to be only temporary.

2. Definition and Environment for Exfoliation Corrosion

Exfoliation corrosion, also referred to as layer, stratified or lamellar corrosion is defined by Lifka and Sprowls (3) as "A particular form of corrosion resulting from a rapid lateral attack along a selective, electrochemically anodic path, parallel to the metal surface. - - - This directional attack results in a leafing action aggravated by voluminous corrosion products that cause uncorroded strata to be split apart." Therefore, the three prerequisites necessary to cause exfoliation corrosion are a preferential anodic corrosion path, a markedly directional grain structure (grains elongated and thin compared to width), and a sufficiently aggressive corrosive environment.

Chloride ions from seawater, bromide ions from combustion products of gasoline aircraft engines, acidic deposits, flue gases and industrial fumes (3,11) were all found to be important chemical factors in an environment aggressive enough to cause exfoliation corrosion in aluminum alloys. It was also determined that $AlCl_3$ was the chief corrosion product. Under total immersion conditions $AlCl_3$ is soluble. However, when an environment permits intermittent drying, insoluble hydrates are formed which exert considerable pressure and result in delamination.

B. Effect of Quench Rate and Aging Procedures on Exfoliation Corrosion

Many investigators (10,12-15) have reported on the effect of quench rate from the solution heat treating temperature on the microstructure of aluminum alloys. In general, there is a precipitate free zone (PFZ) adjacent to grain boundries after aging. The width of this zone is dependent on the solution treating temperature, quench rate and aging temperature. Slower quench rates and higher aging temperatures generally lead to a wider PFZ. It was concluded that the wider the PFZ, the greater the susceptibility to intergranular corrosion. Fink and Willey (10) showed the critical temperature range during quenching of 75S was between 750°-550°F. They found that in this temperature range, quench rates of 800°F/sec or greater were required to achieve the desired strength characteristics. Corrosion resistance was not impaired until a quench rate as low as 200°F/sec existed. At quench rates between 200°F/sec and 120°F/sec pitting was the predominate corrosion mode in NaCl. At quench rates between 120°F/sec and 80°F/sec both pitting and intergranular corrosion occurred. At rates between 80°F/sec and 10°F/sec intergranular corrosion predominated. They concluded that intergranular corrosion resulted from preferential attack of the solid solution adjacent to the grain boundries which is anodic to the material within the grains.

Two theories are presently available to explain the width of the PFZ after heat treatment. The original theory was that the PFZ was an area of solute depletion due to diffusion of the solute to grain boundaries during quenching. Embury and Nicholson (16) maintain the PFZ is an area of vacancy depletion which results from diffusion of vacancies to grain boundaries during quenching rather than solute depletion and propose that it is the vacancy density and distribution which governs the nucleation of precipitate. Investigators (12-19) generally agree on the hardening mechanism in aluminum alloys, i.e., GP zone formation to intermediate precipitate to equilibrium precipitate. Later work by Lorimer and Nicholson (14) and Clark (18) showed that both the solute depletion theory and the vacancy depletion theory are valid and the determination of which mechanism predominates is a function of the aging temperature and the alloy system.

Special aging treatments (5,7,8) or double aging (15,17) treatments tend to equilize the potential difference between the PFZ and the remainder of the grain such that corrosion is more uniformly distributed. This results in less concentrated attack along the grain boundaries and exfoliation corrosion does not occur.

C. Test Methods to Determine Susceptibility to Exfoliation Corrosion

Original methods used to access the susceptibility of an alloy system or condition to exfoliation corrosion were atmospheric exposure tests. These tests were of long duration, extending up to two years sometime to develop the necessary attack. Budd and Booth (20,21) developed a rapid potentiostatic test for assessing susceptibility to exfoliation corrosion. This test was successfully correlated to atmospheric exposure tests, but the area evaluated was so small that metallographic examination was required to assess the results.

An accelerated acidified salt spray test was developed by Lifka and Sprowls (3) to determine susceptibility to exfoliation corrosion. This test consists of exposure to a spray of 5% NaCl solution buffered to pH3 with acetic acid, applied in cycles and followed by drying cycles. The drying cycles allow the corrosion products to dry and to exert pressure which produces delamination. The test is run at elevated temperatures, 95°-120°F, and exposure time is generally from two to four weeks.

A much more severe test for producing exfoliation corrosion has recently been developed (22, 23). This test consists of a continuous spray of 5% NaCl with periodic additions of SO_2 gas to the exposure chamber. The test is run at 95°F for about two weeks. This test has produced exfoliation corrosion in specimens which showed no exfoliation corrosion when exposed to the acidified salt spray tests (23).

D. Thermal-Mechanical Processing of Aluminum Alloys

It has been reported (3) that fabrication techniques which affect the grain shape of a product can affect its exfoliation corrosion resistance. Lifka, Sprowls and Kaufman (6) report that 7178-T6 aluminum alloy heavy plate, 0.150 inches to 0.750 inches in thickness, has a fabrication sequence which causes the recrystallized texture (grain shape) to be optimized for the development of exfoliation corrosion. Plate within that thickness range was therefore selected for evaluation of the thermal-mechanical processing techniques which were employed during this study.

Metcalfe (11) exposed specimens of aluminum-magnesium-silicon and aluminum-copper-magnesium-silicon to a variety of industrial, seacoast, and rural atmospheres and evaluated their effect on the intergranular corrosion resistance after 3,6,12,18 and 24 months. He also plastically deformed some specimens 5% from each alloy system. For the first 6 months the plastically deformed specimens of aluminum-magnesium-silicon had better resistance to intergranular corrosion than the undeformed specimens. Between 6 months and 18 months the corrosive attack on the deformed specimens accelerated and was more severe than on the undeformed specimens. The corrosive attack on the deformed and undeformed specimens of aluminum-copper-magnesium-silicon was the same.

Embury and Nicholson (16) maintain that plastic deformation prior to aging can affect the precipitation pattern in the PFZ since the plastic deformation will affect the vacancy concentration and distribution. Rosenbaum and Turnbull (13) showed that cold work prior to aging resulted in formation of precipitates close to grain boundaries. They maintain that the cold work increases the number of nuclei and thus reduces the width of the PFZ. Benedyk (24) plastically deformed an aluminum-magnesium-silicon alloy 90% and then double aged the specimens. Significant strength increases resulted which he attributed to increased precipitation resulting from the additional number of nucleation sites created by the plastic deformation.

McEvily, Snyder and Clark (25) working with an aluminum -10% magnesium alloy showed that cold reductions of 5% and 10% prior to aging had no effect on the precipitation pattern or width of the PFZ. A 50% cold reduction prior to aging eliminated the PFZ, and caused precipitation along grain boundaries. The mode of fatigue failure changed from intergranular to transgranular in the 50% cold reduced material. However, the plastic deformation and the distribution of precipitates was not uniform.

McEvily, Clark and Bond (9) working with a basic 7000 series aluminum alloy system (Al-5.5Zn-2.5Mg) showed that a 50% cold reduction markedly increased the alloy's resistance to stress corrosion cracking and had a small beneficial effect on the fatigue strength. The cold work induced additional nucleation sites adjacent to grain boundaries and strengthened the grain boundaries by work hardening and creation of jogs along the boundaries.

III. DISCUSSION

A. Experimental Procedure

1. Material Selection

Commercially available 7178 aluminum alloy was used for this evaluation. Plate material in thicknesses of 0.390 and 0.250 inches was selected because it was reported (6) that heavy plate in this thickness range had a fabrication sequence which optimized the grain texture for the development of exfoliation corrosion. The chemical composition required by the procurement specification for this material and as determined by spectrographic analysis is shown in Table I.

The starting material consisted of seven panels 7 inches wide and 12 inches in length of the 0.250 inch thick stock and eight panels of the same dimensions of the 0.390 inch thick stock. All material was in the annealed condition.

2. Processing Prior to Reduction

Three panels of each thickness were solution heat treated in a sodium and potassium nitrate salt bath by heating to $870^{\circ}F \pm 10^{\circ}F$, holding at temperature for one hour and quenching in $70^{\circ}F$ water prior to mechanical processing. One panel of each thickness was left in the annealed condition to serve as a control panel to be heat treated along with the panels which were cold worked. One panel, 0.390 inches thick was maintained in the annealed condition through all testing.

3. Mechanical Deformation

Three annealed and three solution heat treated panels were then reduced by passing through a rolling mill to obtain the desired amount of reduction. One panel of each condition and of each thickness was reduced 10%, 25% and 40% of its original thickness. The rolling mill

TABLE I

CHEMICAL COMPOSITION - 7178 ALUMINUM ALLOY

TEST SPECIMENS

Element	Specification Value QQ-A-250		Test Material	
	Min.	Max.	0.250 Inch Thick	0.390 Inch Thick
Zinc	6.3	7.3	6.5	6.6
Magnesium	2.4	3.1	2.55	2.7
Copper	1.6	2.4	1.75	1.65
Manganese	-	0.30	0.08	0.07
Chromium	0.18	0.40	0.25	0.19
Iron	-	0.70	0.26	0.24
Silicon	-	0.50	0.11	0.11
Titanium	-	0.20	0.03	0.03
Others (Each)	-	0.05	-	-
Others (Total)	-	0.15	-	-
Aluminum	-	Bal.	Bal.	Bal.

employed is shown in Figure 1, and consisted of (a) two high strength polished rolls, (b) a rigid frame to support the rolls, (c) a motor to drive the rolls, (d) a motor-generator power supply, and (e) electrical control instruments for the drive motor. Frictional forces which develop between the specimen and the rolls in a mill having sufficient rigidity and horsepower pull the specimen between the rolls. When these forces develop and the specimen is thicker than the gap between the rolls, the specimen is plastically deformed by the rolls to a thickness which is approximately equal to the height of the gap.

The specimens cold rolled in the annealed condition are identified with an A prior to the specimen number. The specimens reduced in the solution heat treated condition are identified with a W prior to the specimen number. The 0.250 inch thick starting stock was identified by means of an X following the condition code and specimen number.

The reduction schedule and the number of rolling passes required to obtain the desired thickness is shown in Table II.

4. Processing Subsequent to Mechanical Deformation

After cold working, the material reduced in the annealed condition was solution heat treated at 870°F for one hour and water quenched. It was desired to age both sets of material to the high strength condition. For the material cold reduced in the annealed condition and solution heat treated after rolling, this would be the T6 condition. The T6 condition is achieved by aging at 250°F $\pm$ 10°F for 24 to 28 hours. For the material cold reduced in the solution heat treated condition, the high strength condition would be the T8 condition for which no aging schedule was available.

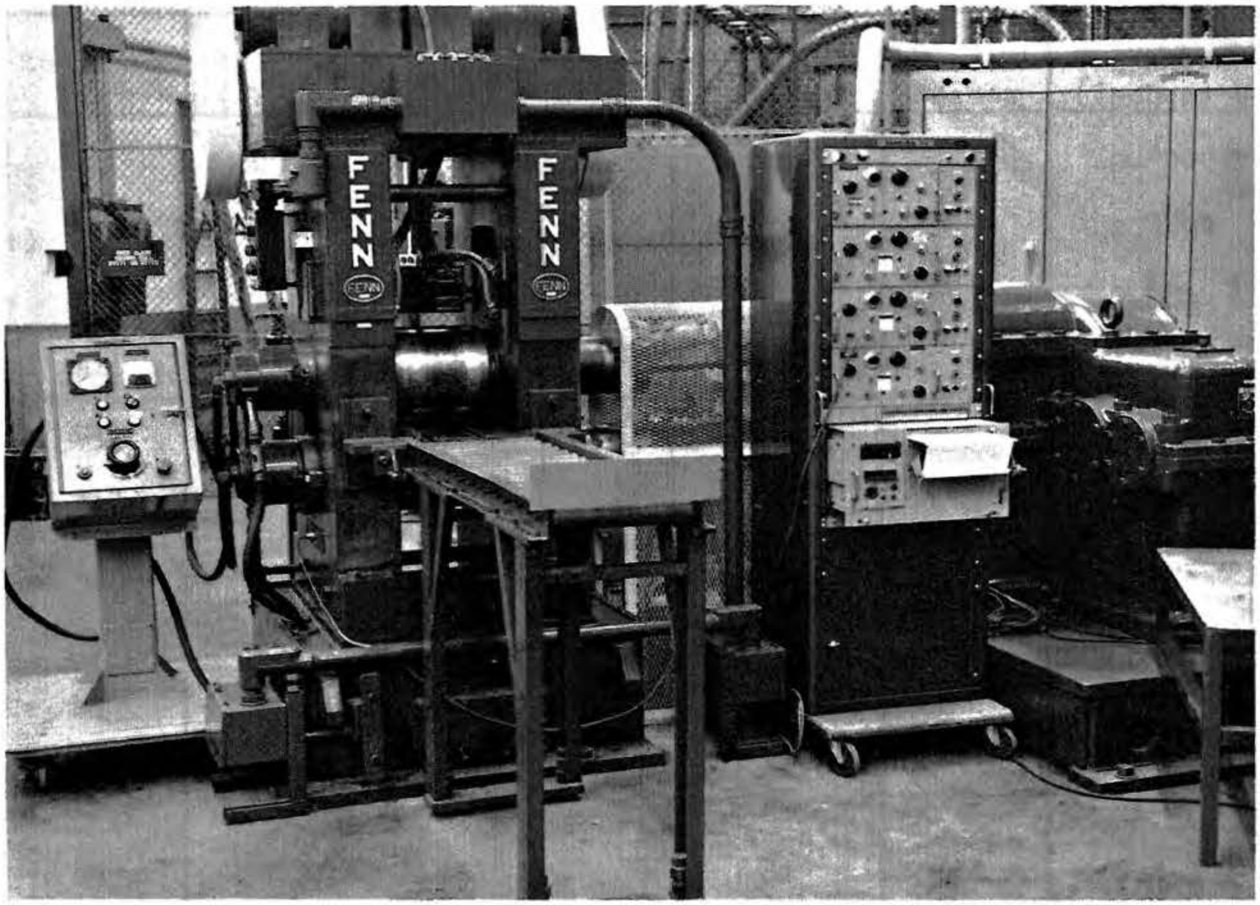


FIGURE 1 – Rolling Mill Used For Mechanical Deformation

TABLE II

REDUCTION SCHEDULE

Specimen No.	Initial Thickness (Inches)	Condition	Reduction (%)	No. of Passes	Final Thickness (Inches)
A4	0.390	Annealed	0	0	0.390
A2			10	1	0.349
A3			25	3	0.281
A1			40	5	0.241
W4X	0.250		0	0	0.250
A4X			10	1	0.225
A3X			25	2	0.187
A2X			40	3	0.147
W1	0.390	Solution Heat Treated	10	2	0.352
W3			25	5	0.297
W4			40	9	0.239
W2X	0.250		10	1	0.226
W3X			25	3	0.189
W1X			40	5	0.149

Therefore, twenty, one inch by one inch specimens were cut from each of the test panels including the control panels of each thickness. Each of the twenty test blanks from each panel and condition were then placed in an electrically heated aging furnace operating at $250^{o}F \pm 5^{o}F$. One specimen from each condition was removed from the furnace after 1, 2, 3, 4 and 5 hours of aging. After the first 5 hours of aging, specimens were removed from the furnace every two hours, up to 27 hours of aging time. The Rockwell "B" hardness and electrical conductivity, as measured in percent of the International Annealed Copper Standard (%IACS) was recorded for each specimen. The hardness and electrical conductivity are plotted versus aging time in Figure 2 thru Figure 15 The electrical conductivity was determined by means of a Magnatest FM-100 electrical conductivity unit.

This approach was taken to develop an aging schedule for the material cold reduced in the solution heat treated condition. This material would have a more rapid aging response due to the cold work. The criteria used to select the proper aging time was to select an aging time where the hardness and electrical conductivity of the T8 specimens was comparable to the hardness and electrical conductivity of the T6 specimens.

An aging time of 17 hours at $250^{o}F$ was selected to produce the T8 condition.

The remainder of each panel was then aged for the required length of time to produce the high strength condition.

After aging, two, three inch by six inch exfoliation corrosion test specimens were cut from each panel. A minimum of two tensile specimens were also prepared from each panel. The results of the tensile tests are shown in Table III.

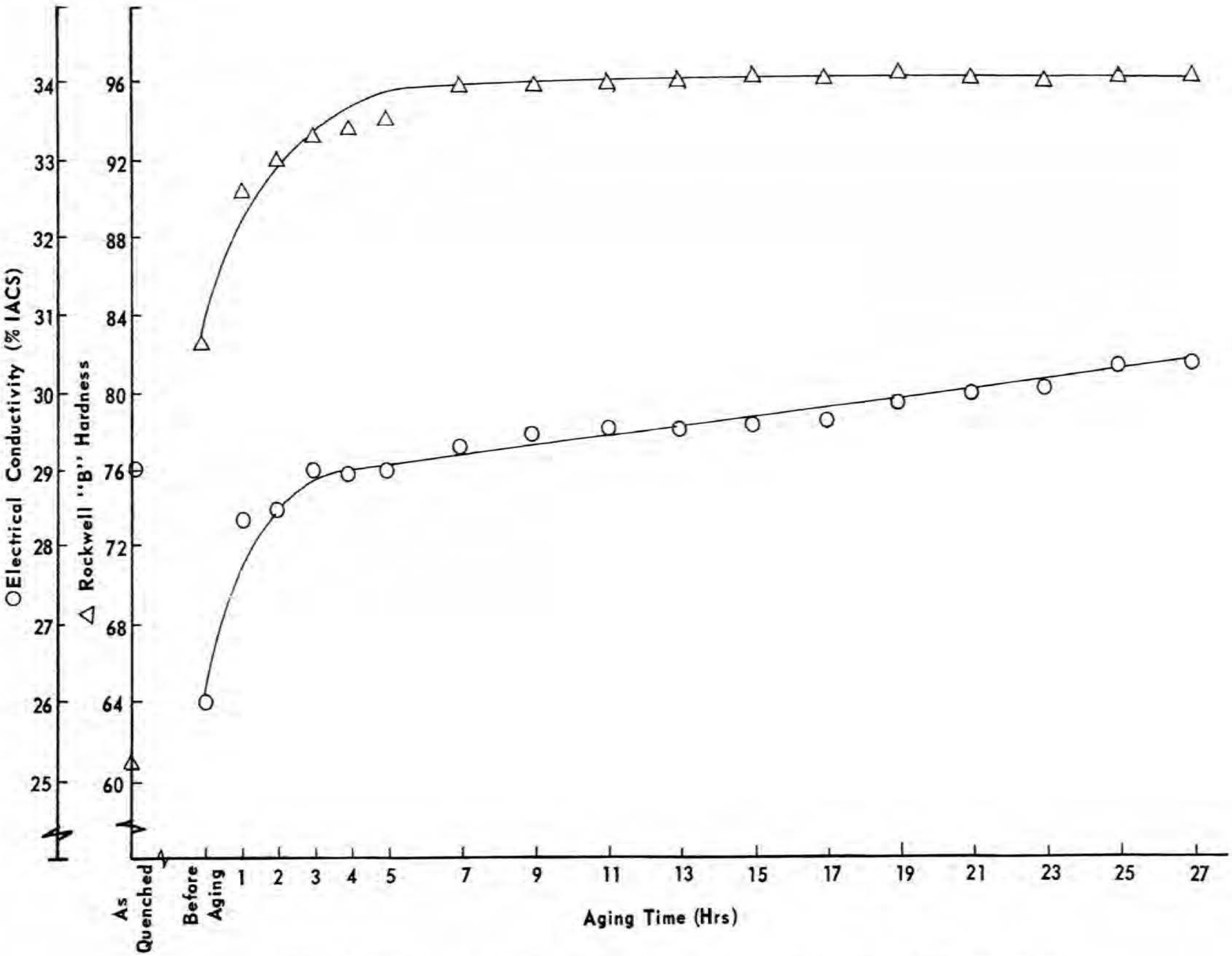


FIGURE 2 – Hardness and Electrical Conductivity versus Aging Time for Specimen A4

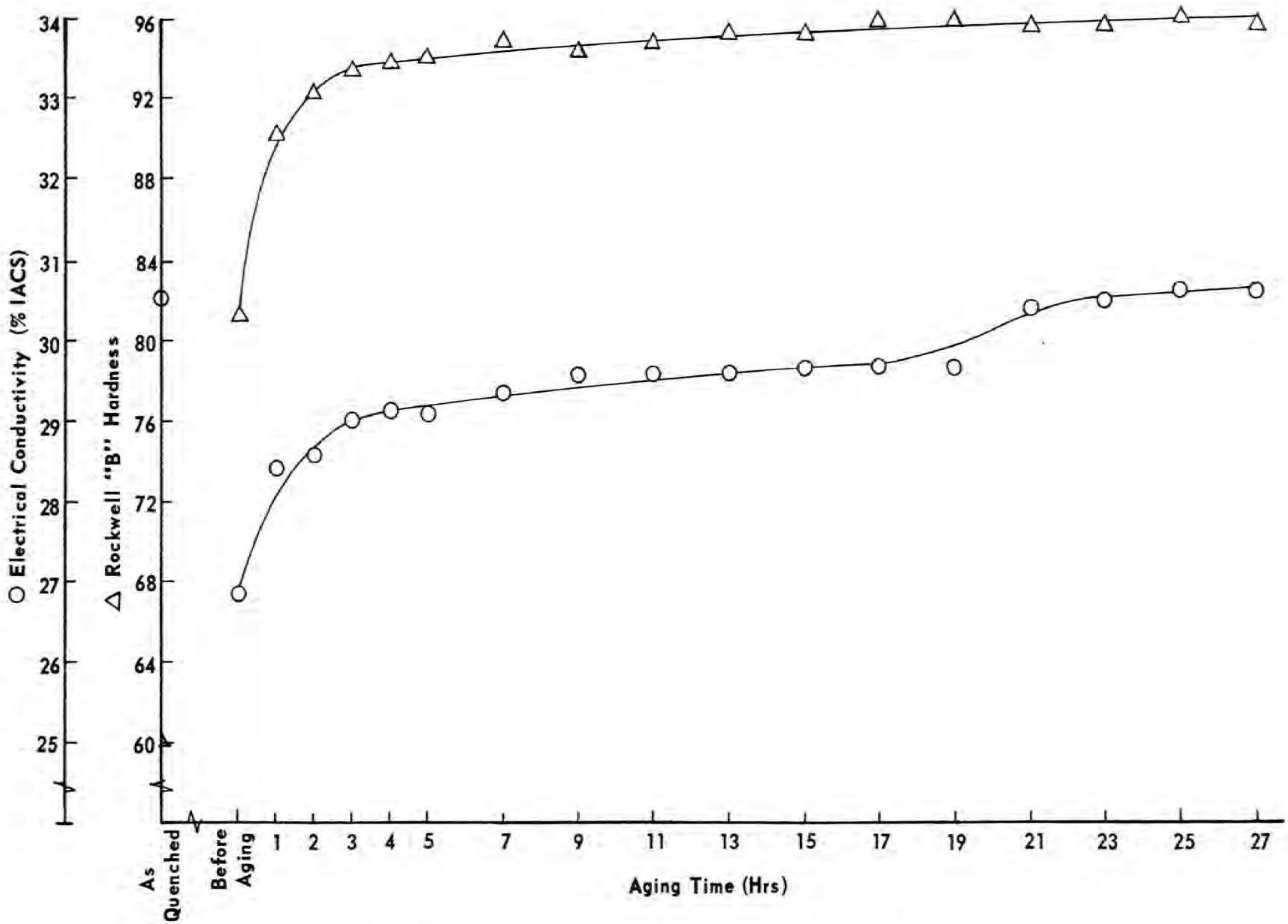


FIGURE 3 – Hardness and Electrical Concuctivity versus Aging Time for Specimen A2

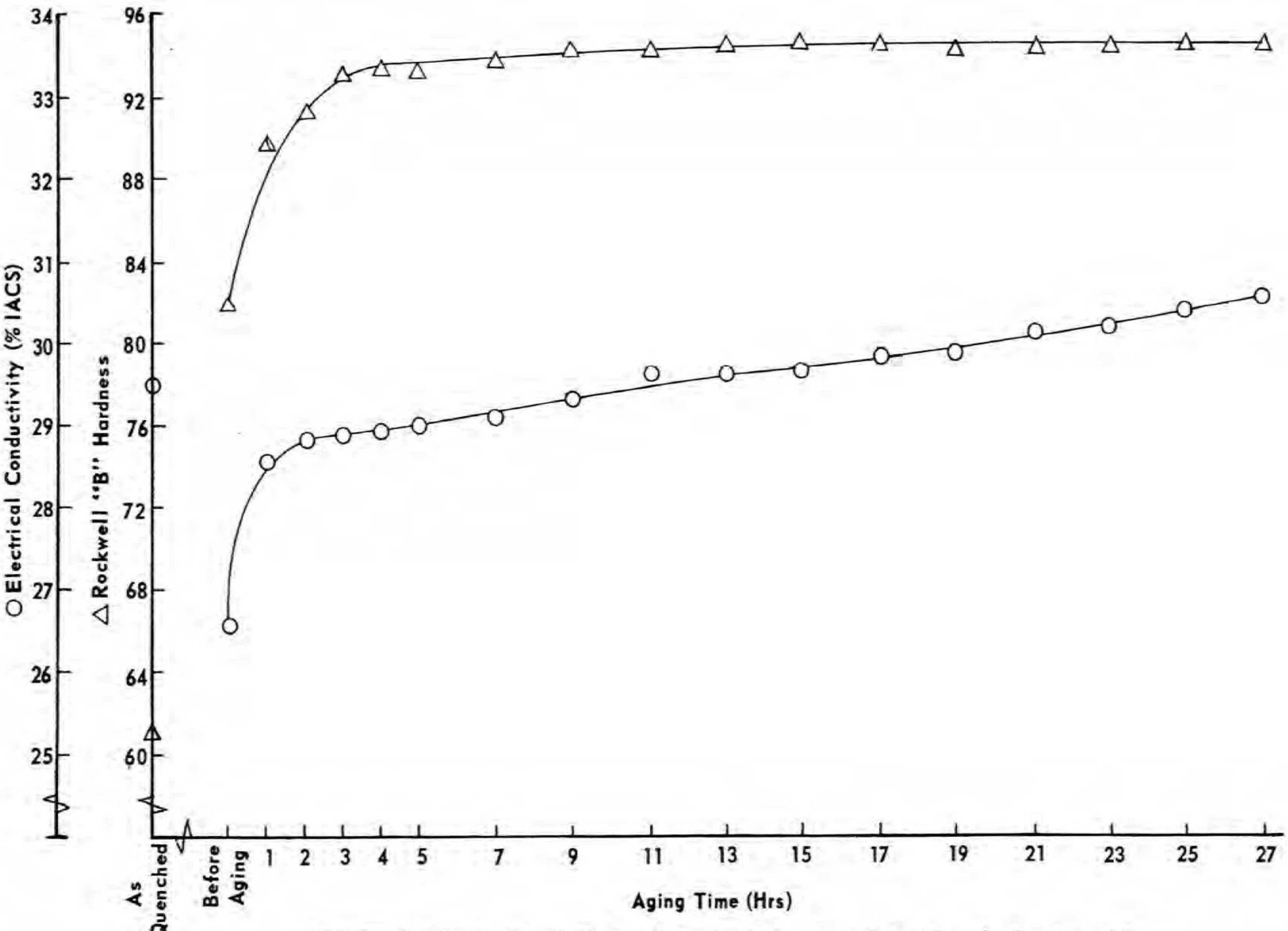


FIGURE 4 – Hardness and Electrical Conductivity versus Aging Time for Specimen A3

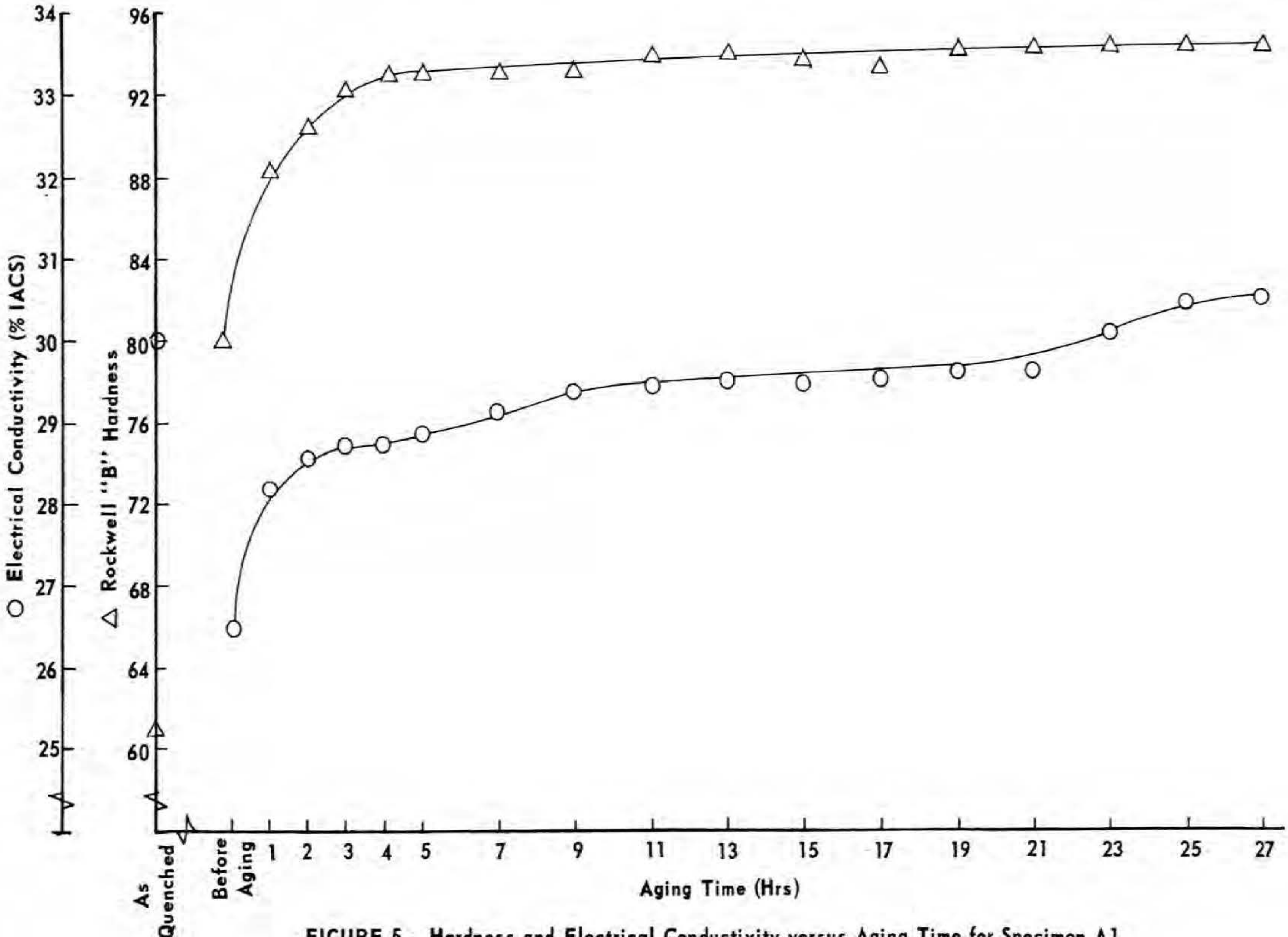


FIGURE 5 – Hardness and Electrical Conductivity versus Aging Time for Specimen A1

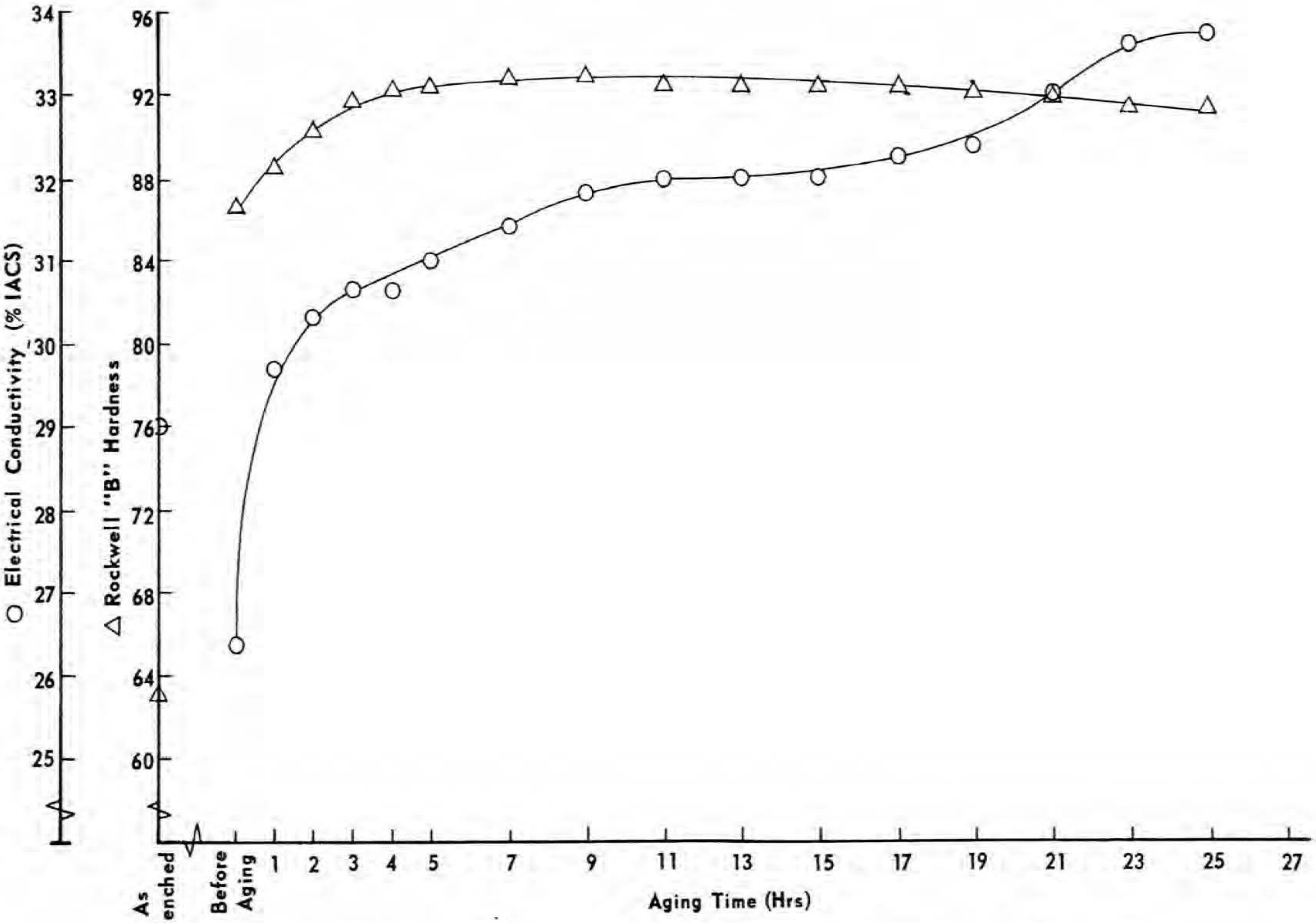


FIGURE 6 – Hardness and Electrical Conductivity versus Aging Time for Specimen W1

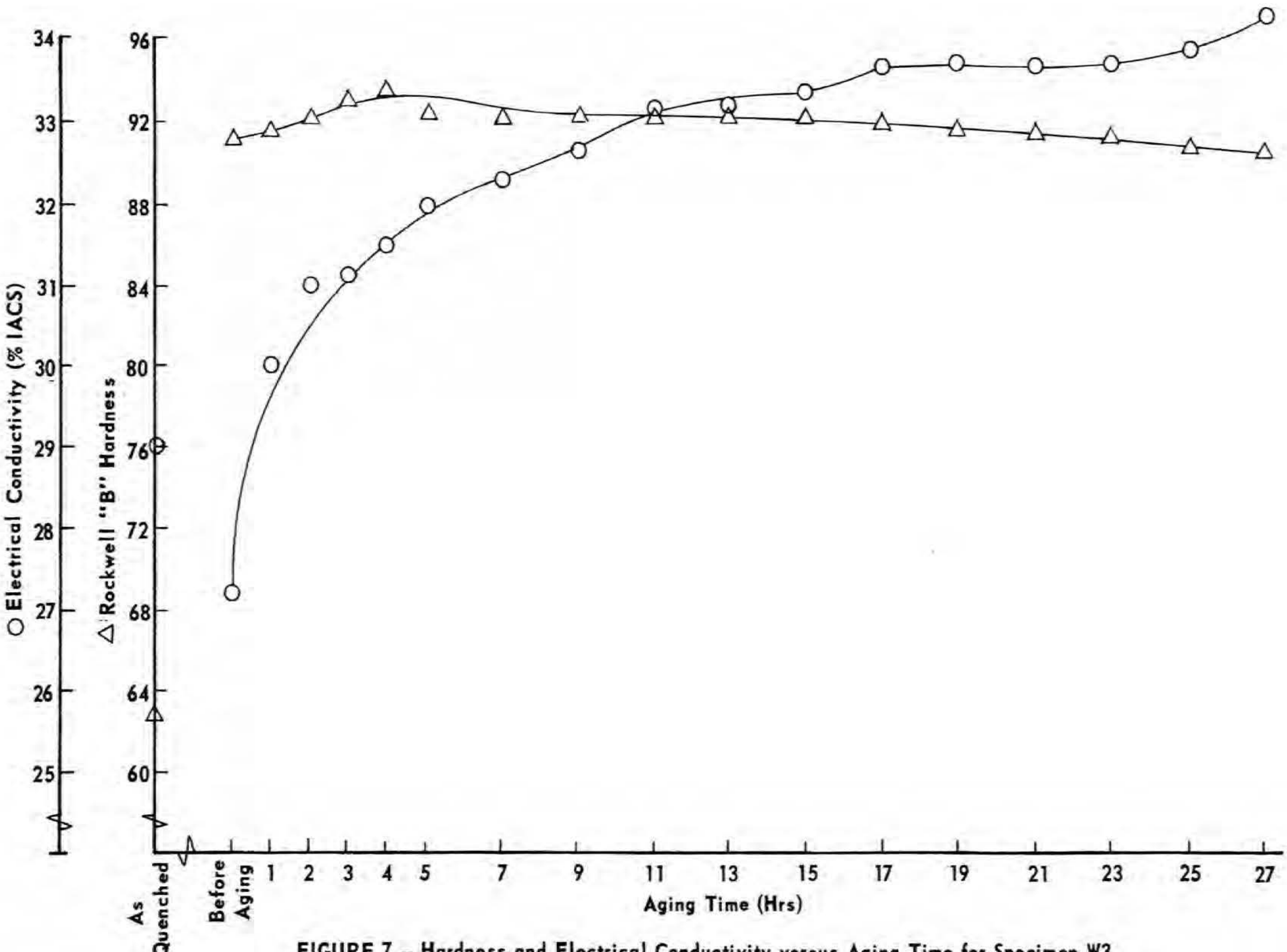


FIGURE 7 – Hardness and Electrical Conductivity versus Aging Time for Specimen W3

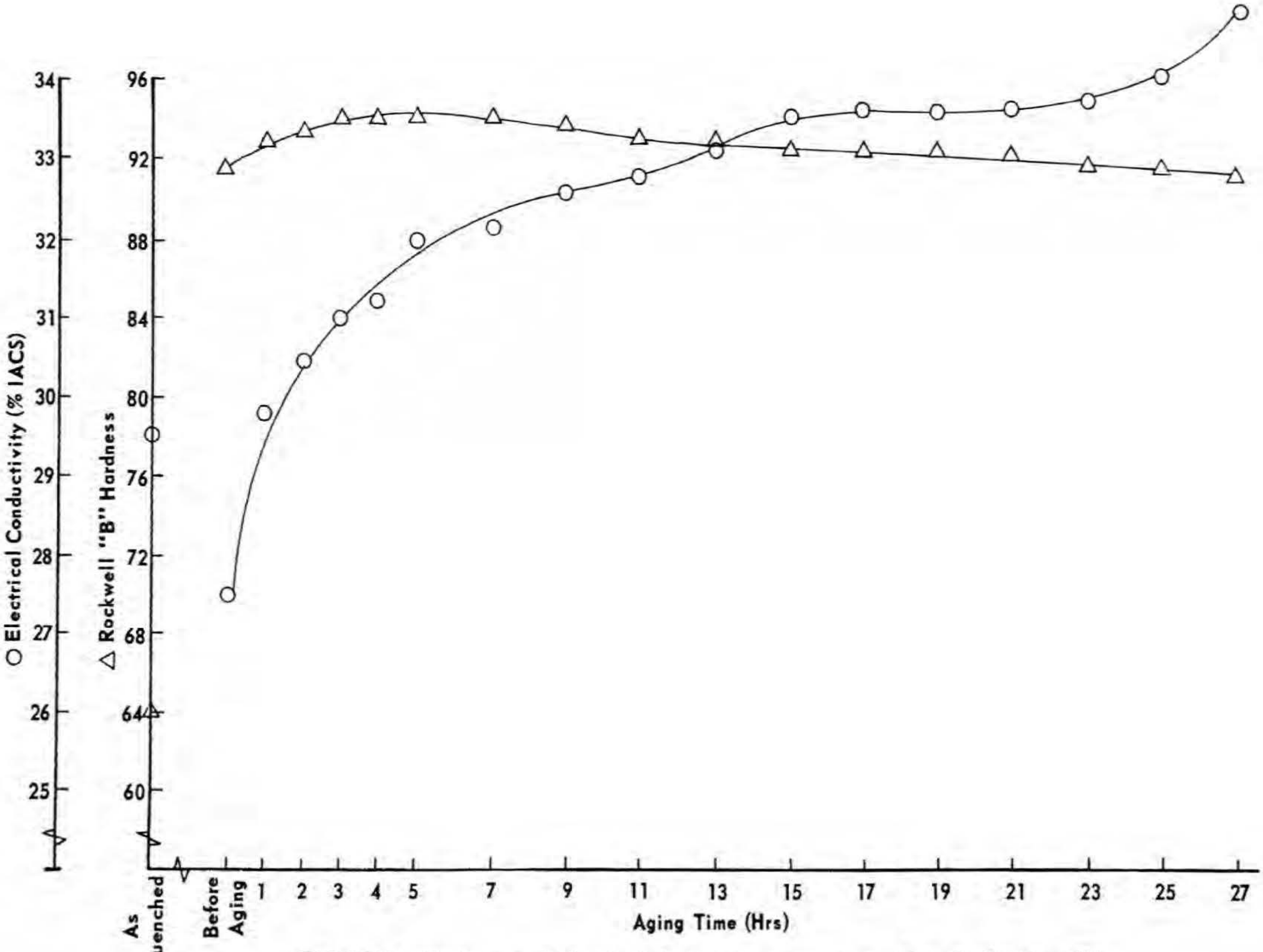


FIGURE 8 – Hardness and Electrical Conductivity versus Aging Time for Specimen W4

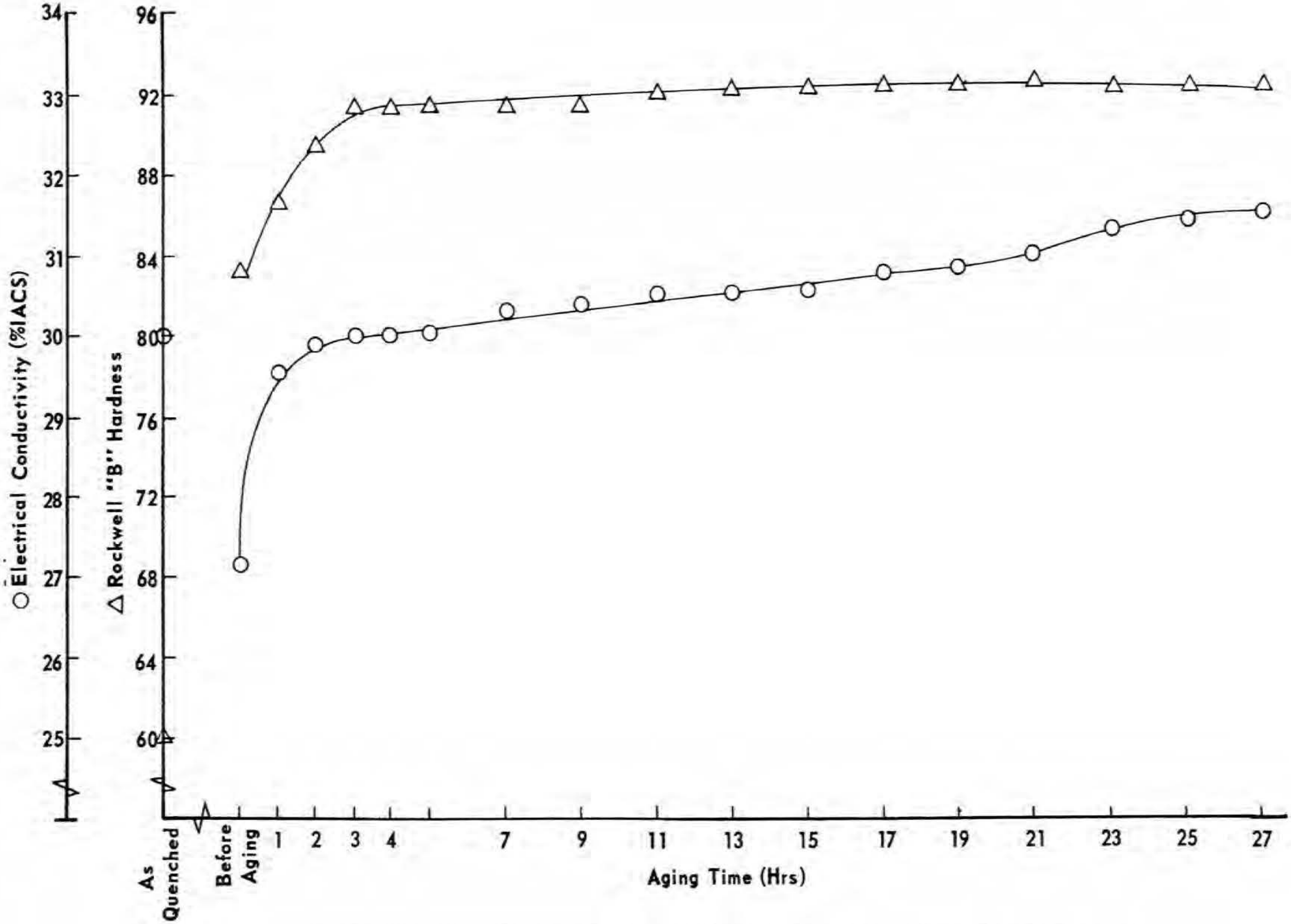


FIGURE 9 – Hardness and Electrical Conductivity versus Aging Time for Specimen W4X

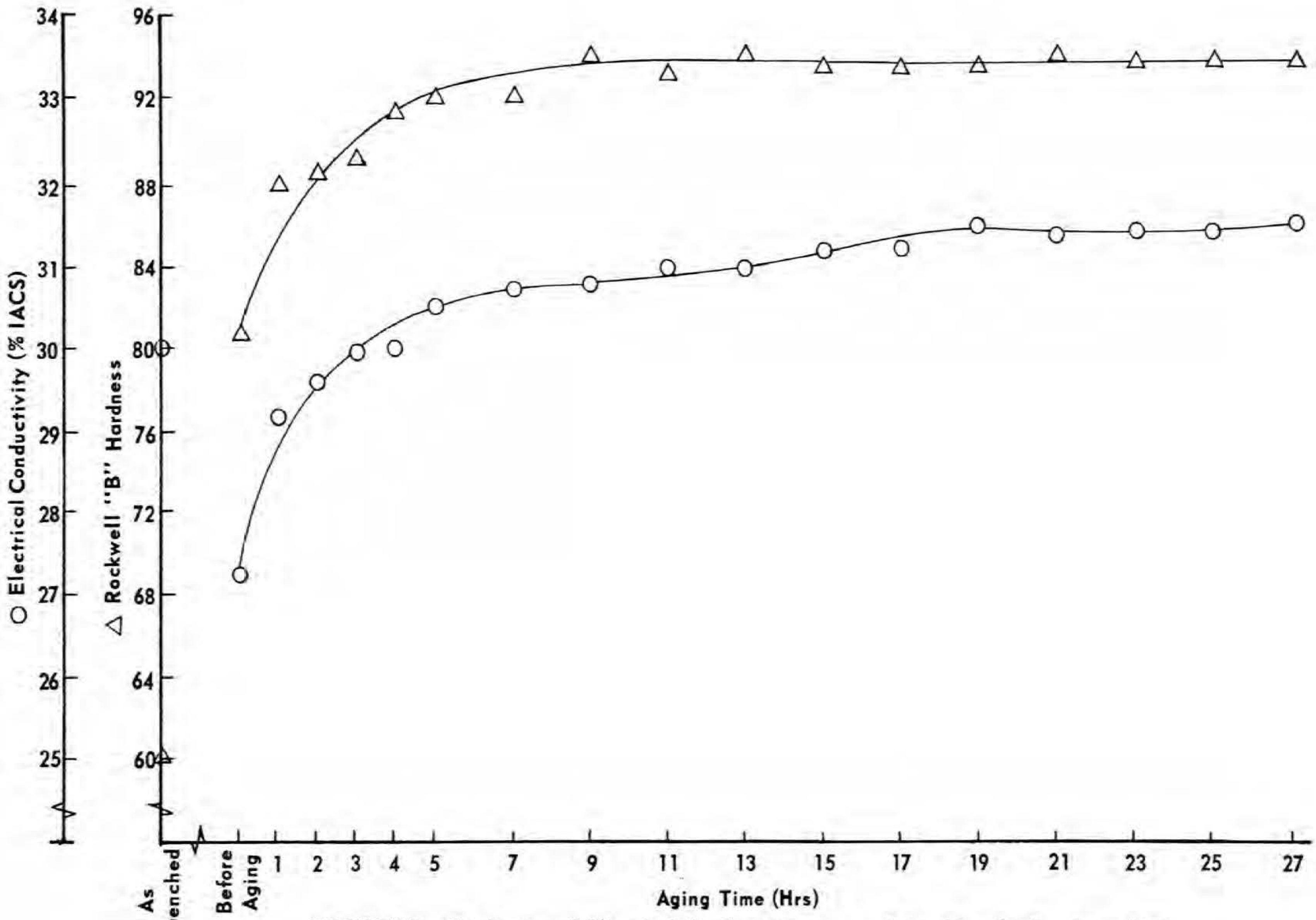


FIGURE 10 – Hardness and Electrical Conductivity versus Aging Time for Specimen A4X

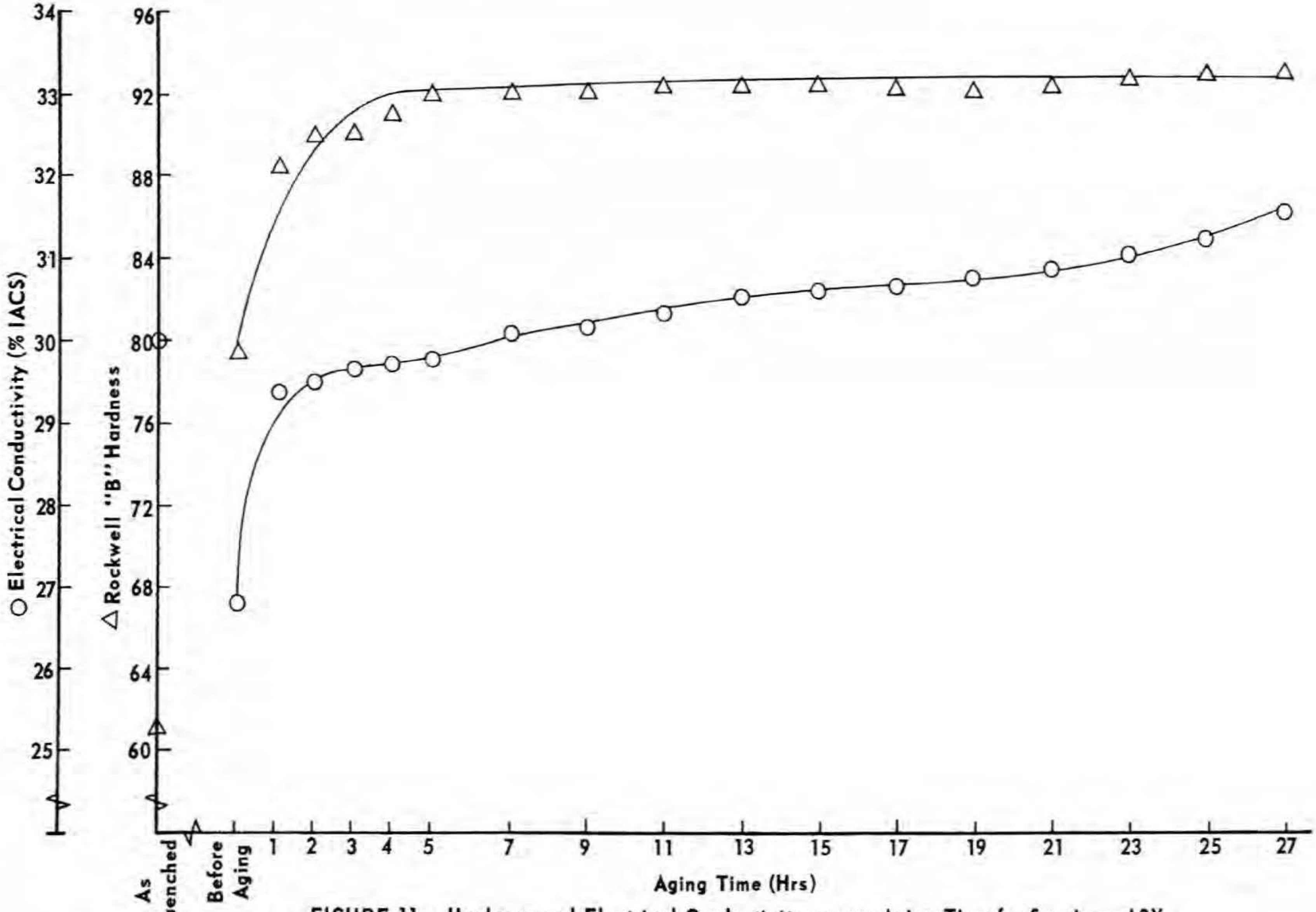


FIGURE 11 – Hardness and Electrical Conductivity versus Aging Time for Specimen A3X

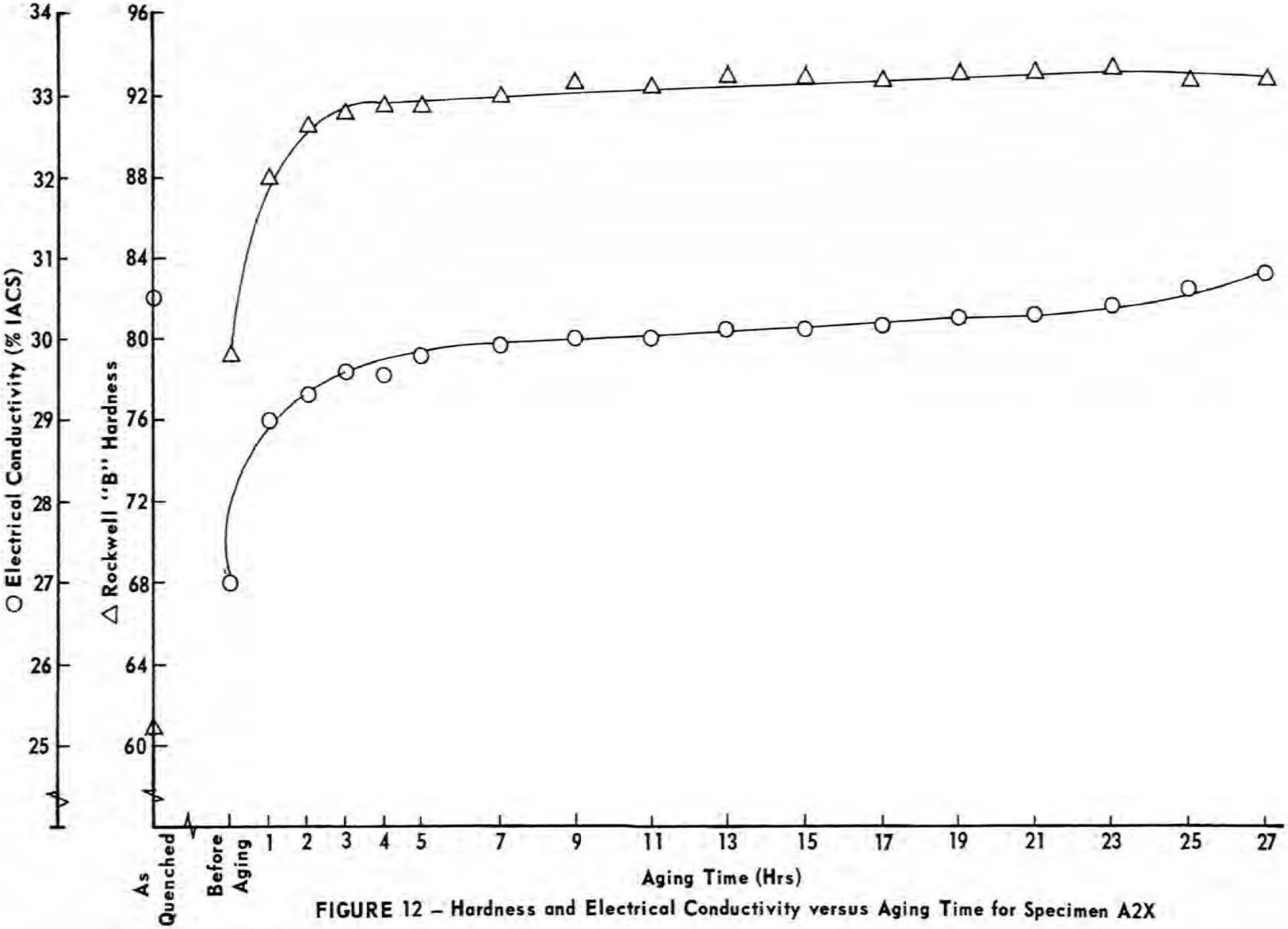


FIGURE 12 – Hardness and Electrical Conductivity versus Aging Time for Specimen A2X

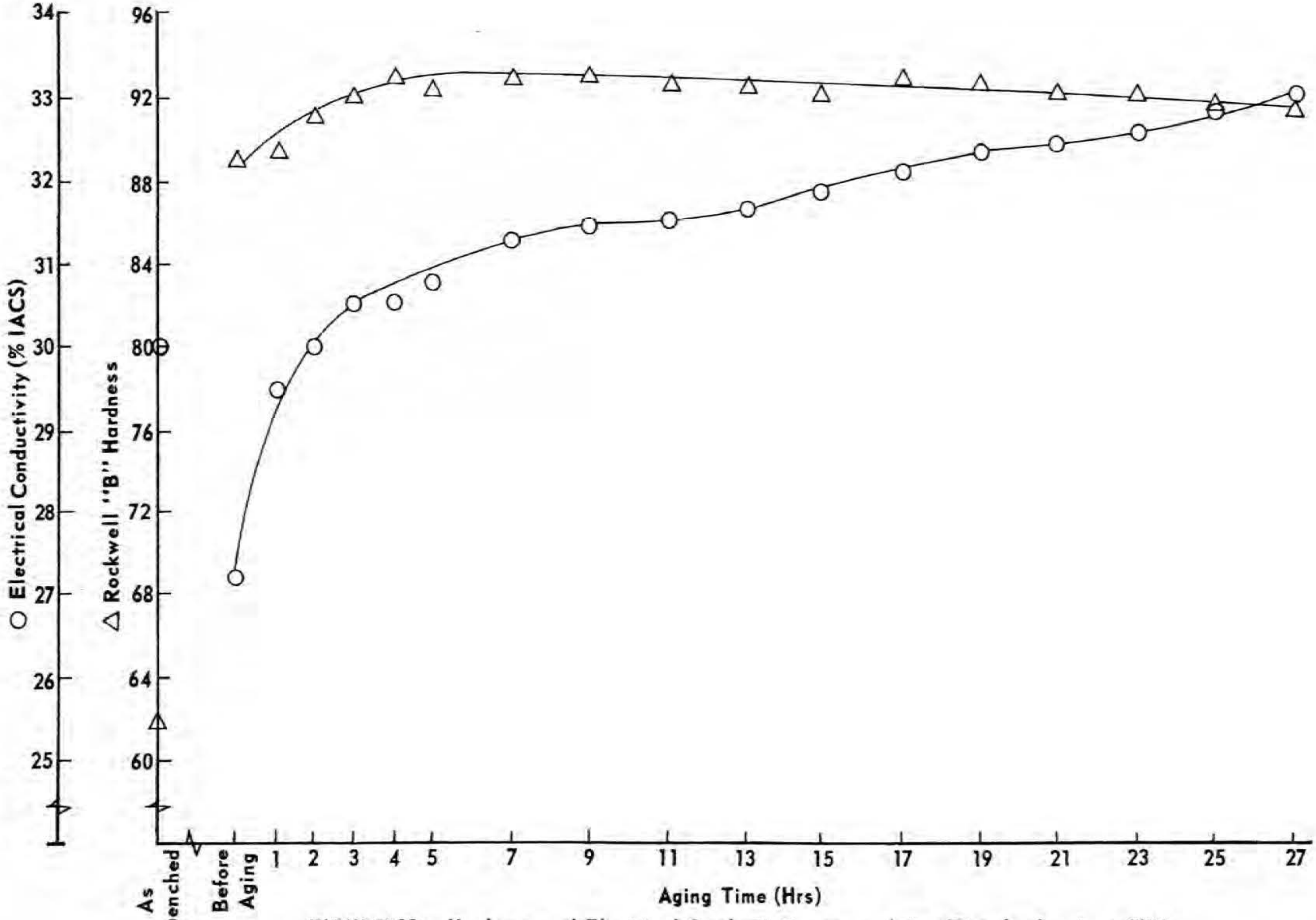


FIGURE 13 – Hardness and Electrical Conductivity versus Aging Time for Specimen W2X

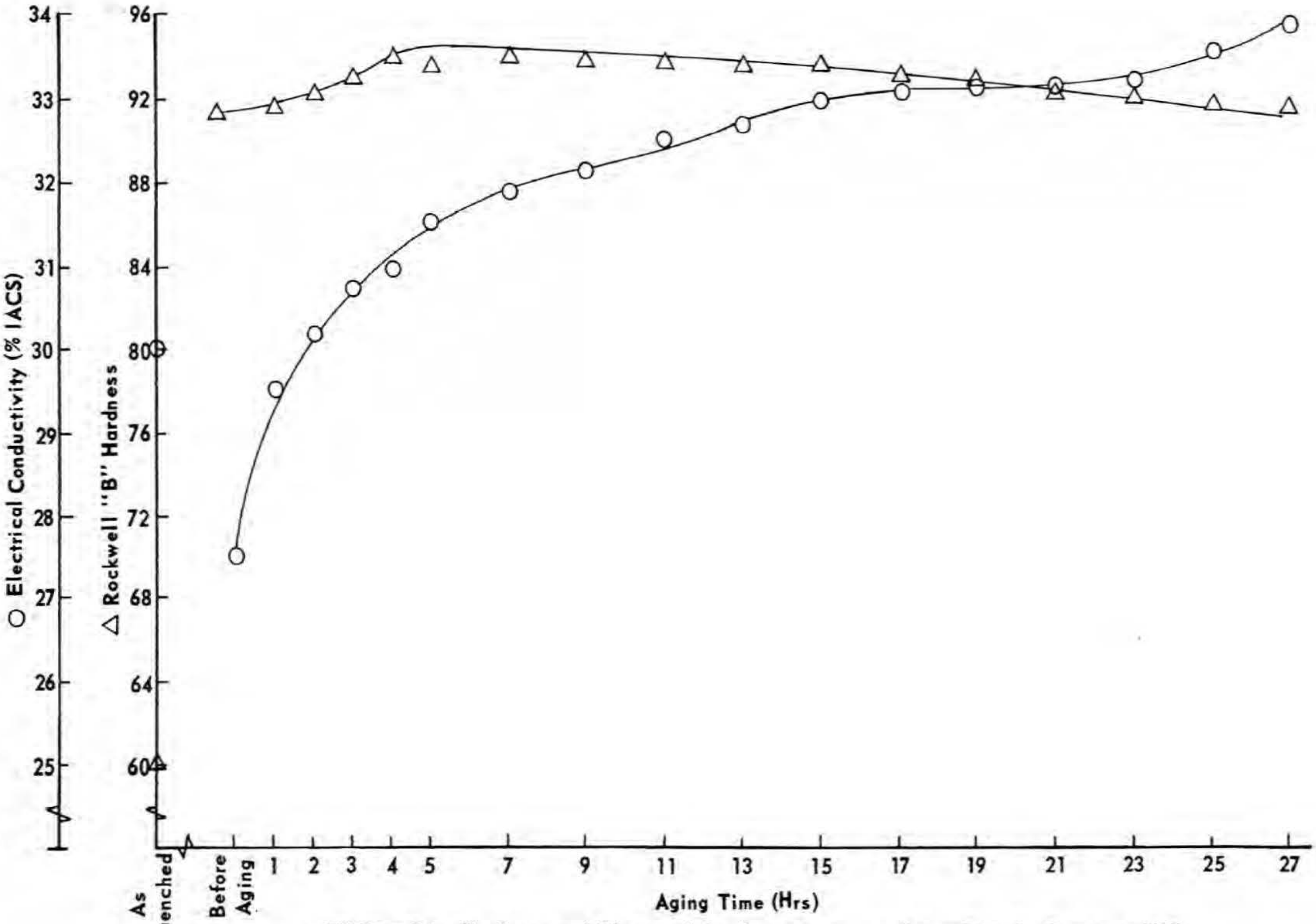


FIGURE 14 – Hardness and Electrical Conductivity versus Aging Time for Specimen W3X

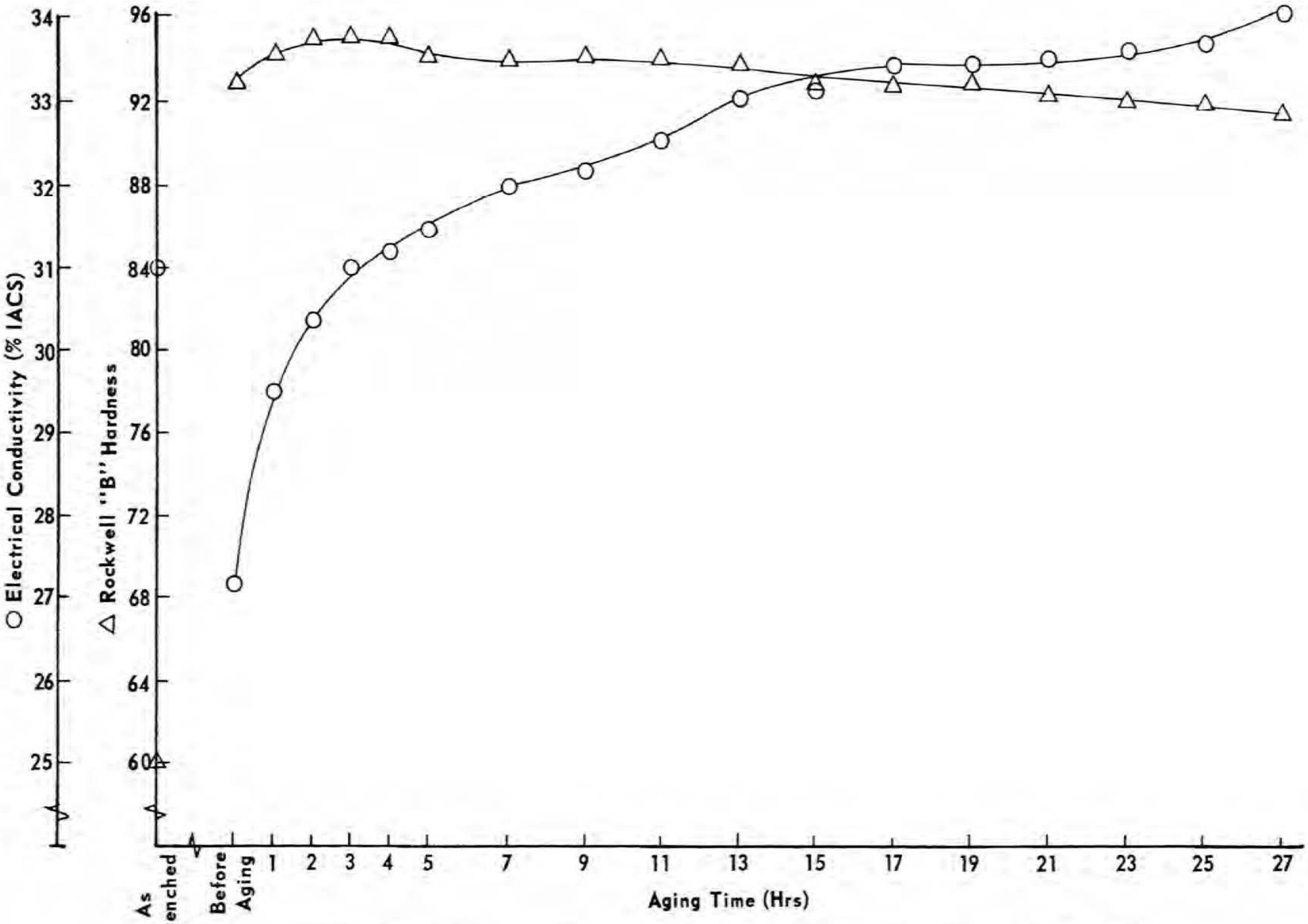


FIGURE 15 – Hardness and Electrical Conductivity versus Aging Time for Specimen W1X

TABLE III

MECHANICAL PROPERTIES OF PROCESSED SPECIMENS

Specimen No.	Reduction (%)	Ultimate Tensile Str. (KSI)	Yield Tensile Strength (KSI)	Elongation in 2 Inches (%)
Specification Minimum	-	78.0	68.0	8
A4	0	88.6	80.8	12.5
A2	10	87.9	81.4	11.7
A3	25	89.6	80.5	15.0
A1	40	89.1	80.0	13.0
W1	10	88.8	80.8	13.0
W3	25	88.6	83.5	11.8
W4	40	85.8	80.8	8.3
W4X	0	87.9	80.0	14.3
A4X	10	85.4	78.1	10.0
A3X	25	86.5	77.4	11.8
A2X	40	86.8	74.1	15.0
W2X	10	86.8	80.4	11.5
W3X	25	85.0	79.8	7.8
W1X	40	86.4	81.0	8.3

5. Exfoliation Corrosion Tests

Each of the three inch by six inch test panels, had one half the length chemically milled in a conventional alkaline chemical milling solution to mid thickness. This was done to expose both surface and midplane grain boundaries to the conditions of the exfoliation corrosion tests. The susceptibility of aluminum alloys to exfoliation corrosion can vary between surface and midplane areas.

The entire specimen was then cleaned in the chem-mill solution before determining susceptibility to exfoliation corrosion by one of the following test methods.

One panel from each of the reduced specimens along with a control panel of both the 0.250 and 0.390 inch stock in the T6 condition and an annealed 0.390 inch thick panel were exposed to the following two exfoliation corrosion test environments.

The first test consisted of exposure of the panels to a 5% sodium chloride solution made with deionized water and adjusted to a pH of 3.0 with acetic acid. The exposure cycle consisted of a 45 minute spray cycle, followed by a two hour air purge of the chamber and a three hour and forty five minute period at 40-90% relative humidity. The temperature in the chamber was maintained at $95^{o}F \pm 5^{o}F$. The specimens were placed in the chamber, as shown in Figure 16, at a 45^{o} angle of inclination with the surface and exposed midplane surface facing upward. Exposure time was 30 days.

The second test was in a more severe environment which consisted of a continuous spray of 5% sodium chloride with periodic addition of sulfur dioxide gas (SO_2) to the chamber. The SO_2 gas was admitted to the 35.7 cubic foot chamber at a flow of 450 cc/minute over two 30

FIGURE 16 – Specimen Orientation During Exfoliation Corrosion Tests

minute periods, starting at 8:00 o'clock a.m. and 1:00 o'clock p.m. each work day, Monday through Friday. During the weekend, the 5% sodium chloride spray was continued without the periodic additions of SO_2 gas. The chamber was continuously purged by forcing air through a bubble tower into the chamber at the rate of 19,000 cc/minute. The pH of collected fog varied between 1.5 and 2.0. The temperature of the chamber was maintained at $95^{o}F \pm 5^{o}F$. The specimens were again placed in the chamber at a 45^{o} angle of inclination with the surface and exposed midplane facing upward. Because of the severity of this test, the specimens were removed after 14 days.

After the exfoliation tests, the corrosion products were removed from the specimens by soaking them for one minute in a 50% solution of nitric acid. The specimens were then rinsed in water, dried and photographed.

6. Metallographic Evaluation Tests

Metallographic specimens were then taken from each corrosion test panel. The specimens were taken so that both the surface and midplane areas could be examined. The metallographic specimens were prepared by rough polishing on silicon carbide paper through 600 grit, followed by polishing on a nylon wheel with diamond abrasive. Final polishing was accomplished using first Linde A polishing abrasive and finishing with magnesium oxide abrasive. The specimens were then etched with Keller's etchant (95% H_2O, 1% HF, 1.5% HCl, 2.5% HNO_3). Examination was performed on a Bausch and Lomb research metallograph. Photomicrographs at 75X were prepared of the most severely exfoliated area on both the surface and midplane on each specimen. The microstructure of each specimen was then characterized by preparing a 250X photomicrograph of the structure.

B. Experimental Results

1. General Objectives

The objective of this evaluation was to develop a T8 condition in commercial 7178 aluminum alloy which retained the mechanical properties of the T6 condition but had the improved exfoliation corrosion resistance of the special overaged T76 condition. The reduction schedules of 10%, 25%, and 40% were based on the work of McEvily, Snyder and Clark (25) and McEvily, Clark and Bond (9) which indicated significant amounts of reduction were required to influence the precipitation pattern enough to improve the materials resistance to stress corrosion cracking, a particular form of intergranular corrosion, as is exfoliation corrosion.

2. Development of Aging Procedures

The material which was cold rolled in the annealed condition and then solution heat treated and water quenched, was aged in accordance with the standard schedule of 24 to 28 hours at 250 ± 5°F. The material which was cold rolled in the solution heat treated condition was aged in accordance with a modified aging schedule. This schedule was developed by aging specimens at 250 ± 5°F for various lengths of time and then monitoring the Rockwell hardness and electrical conductivity as measured in % IACS. An aging time of 17 hours was selected to provide a hardness equivalent to the material aged in accordance with the established aging procedures. The aging time of 17 hours also reflected the point immediately prior to the rapid increase in electrical conductivity. It was felt that this point would provide mechanical properties equivalent to the T6 condition. The rise in electrical conductivity subsequent to this point indicates rapid overaging which would result in a reduction in strength, primarily tensile yield strength. Figures 6, 7, 8, 13, 14 and 15 show this rise in electrical conductivity along with a minor decrease in Rockwell hardness. Selection of an aging schedule beyond the 17 hours would have resulted in lower strength, but improved corrosion resistance because the overaging minimizes the potential difference between the grain boundaries, material adjacent to the grain boundries, and the grains themselves. Such a treatment would be similar to the already established overaging schedules used to improve the exfoliation corrosion resistance but which reduce the strength of the material.

3. Mechanical Properties of Cold Reduced Material

The aging treatment selected resulted in mechanical properties which did not vary significantly from the mechanical properties of the material reduced in the annealed condition and solution heat treated and aged using the standard aging time. This is depicted graphically in Figure 17.

Therefore, the first objective of this evaluation was accomplished, i.e., to develop a T8 condition with mechanical properties equivalent to those of the T6 condition.

4. Effect of Exfoliation Corrosion Tests on Annealed 0.390 Inch Thick Material

The 0.390 inch thick annealed material exposed to the two exfoliation corrosion tests showed no exfoliation in the acidified salt spray test but did exhibit a minor amount of pitting at midplane. In the NaCl plus SO_2 environment a very minor amount of exfoliation was

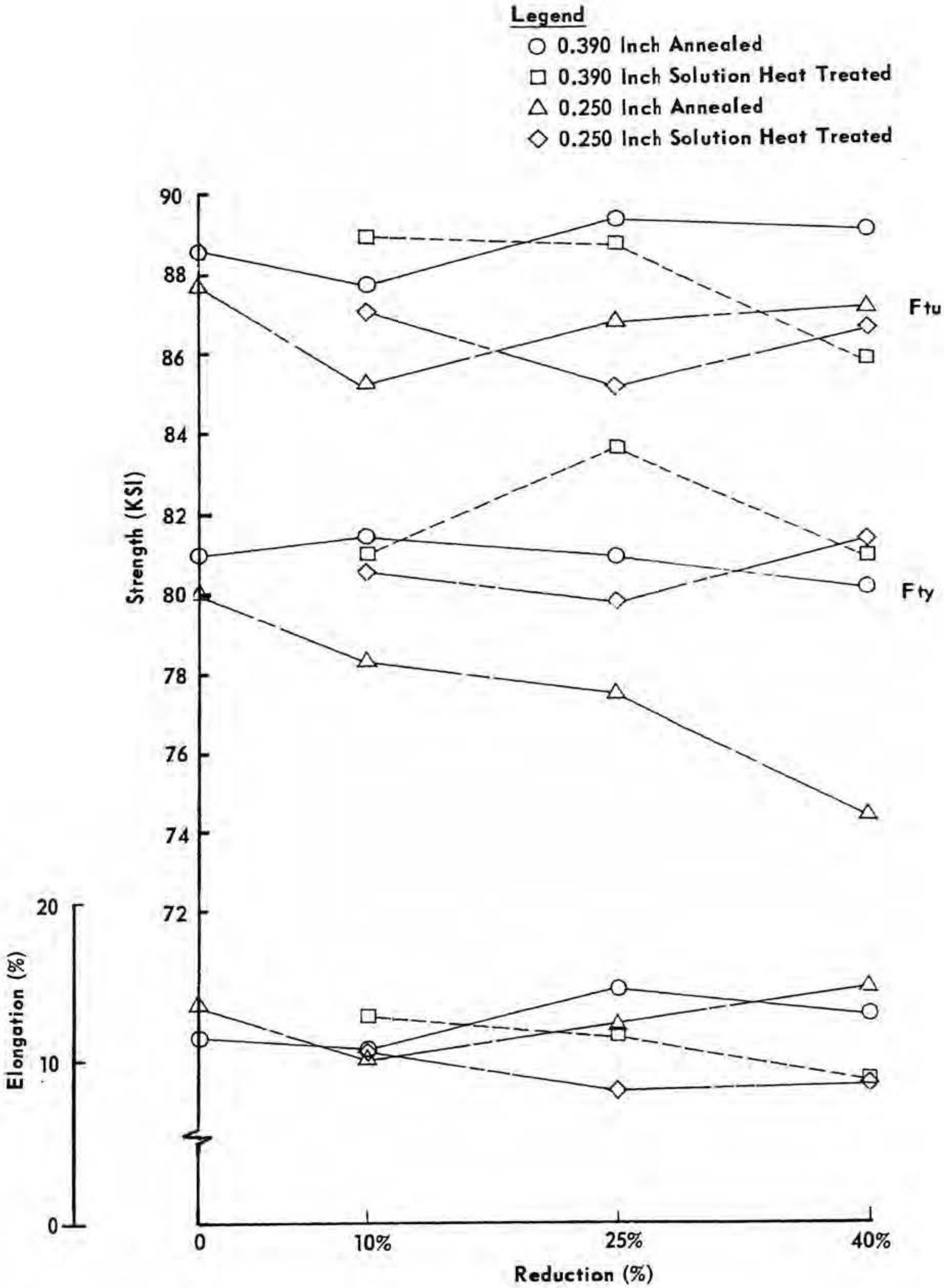


FIGURE 17 – Graph of Mechanical Properties versus Percent Reduction

evident at midplane. Metallographic examination of this specimen after exposure to both corrosion tests is shown in Figure 18.

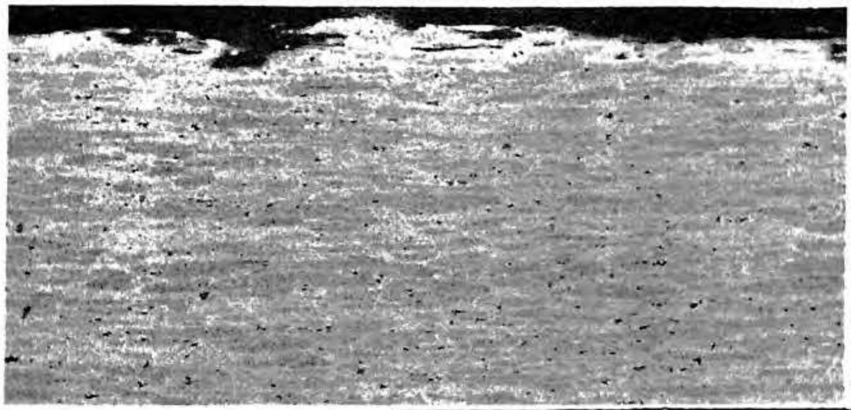
5. Effect of Exfoliation Corrosion Tests on Cold Reduced 0.390 Inch Thick Material

The 0.390 inch thick material control specimen was slightly exfoliated at both the surface and midplane and pitted on both the surface and midplane areas, as illustrated in Figure 19, by the acidified salt spray tests. The NaCl plus SO_2 gas environment produced even greater exfoliation corrosion as demonstrated in Figure 20.

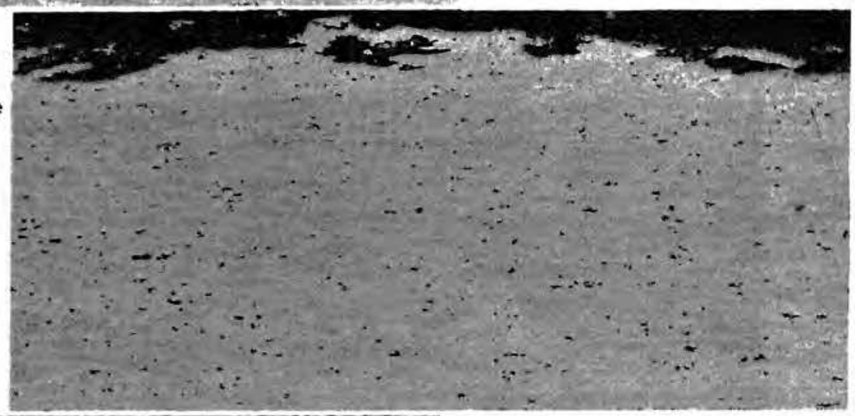
The specimen reduced 10% in the annealed condition and then solution heat treated and aged was pitted slightly more than the control specimen in the acidified salt spray but the exfoliation attack remained about the same. The exfoliation corrosion attack in the NaCl plus SO_2 gas environment was more severe than on the control specimen. Figure 21 illustrates the severity of attack caused by the acidified salt spray and Figure 22, the attack caused by the more severe NaCl plus SO_2 gas test environment.

As the amount of reduction increased to 25% and 40%, the exfoliation corrosion resistance began to improve as demonstrated in Figures 23 through 26. The material reduced 40% in the annealed condition pitted severely in the acidified salt spray but did not exfoliate. A slight amount of exfoliation, less than on the control specimens, was evident on the specimen exposed to NaCl plus SO_2 gas.

The specimens reduced 10% and 25% in the solution heat treated condition and aged after cold reduction exfoliated as severely in the acidified salt spray environment, Figure 27 and 29, as the

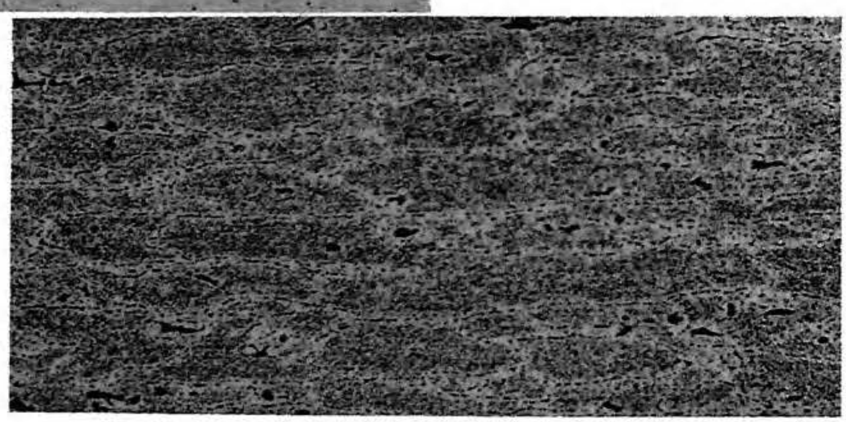


Surface After Acidified Sodium Chloride Corrosion Test
MAG. 75X



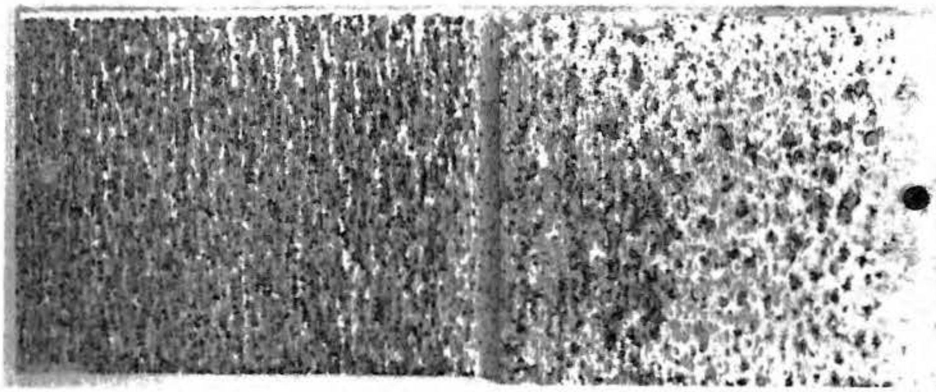
Surface After Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test
MAG. 75X

Midplane After Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test
MAG. 75X

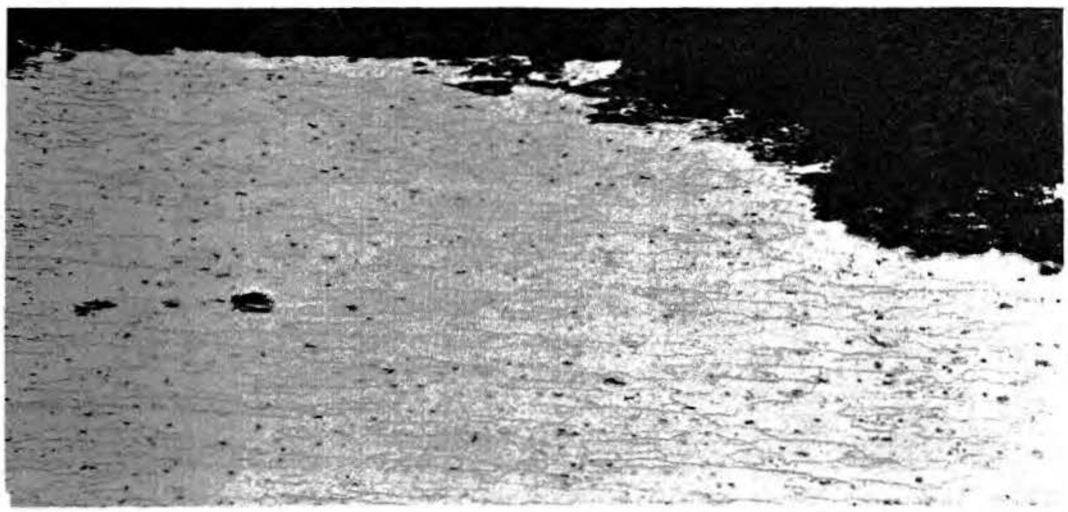


Microstructure of Annealed 0.390 Inch Thick Specimen
MAG. 250X

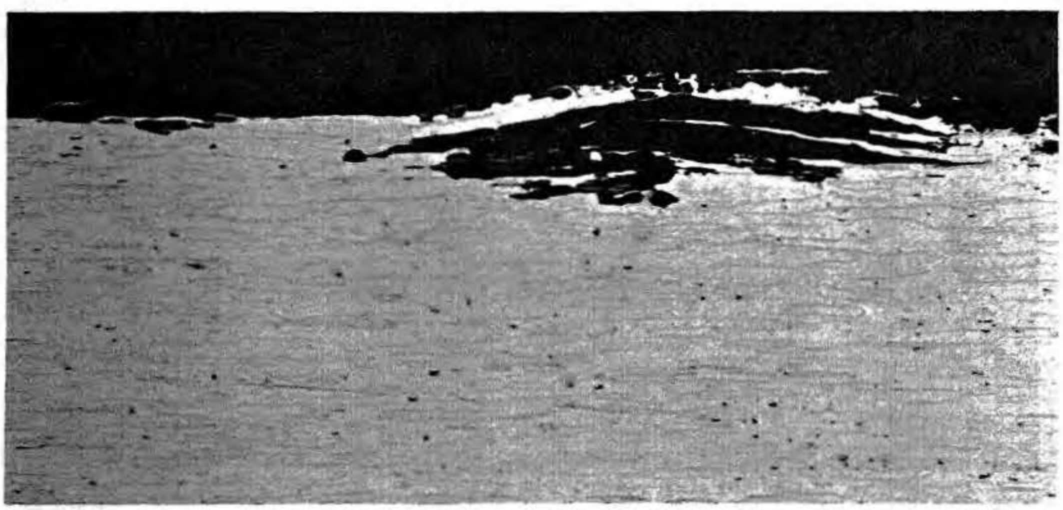
FIGURE 18 – Specimen A5 After Exposure to Both Exfoliation Corrosion Tests



Reduced 30%

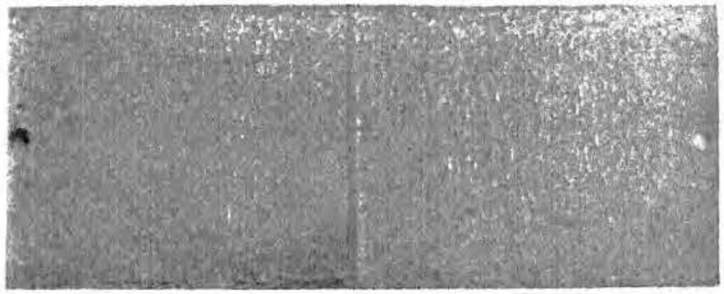


Surface MAG. 75X



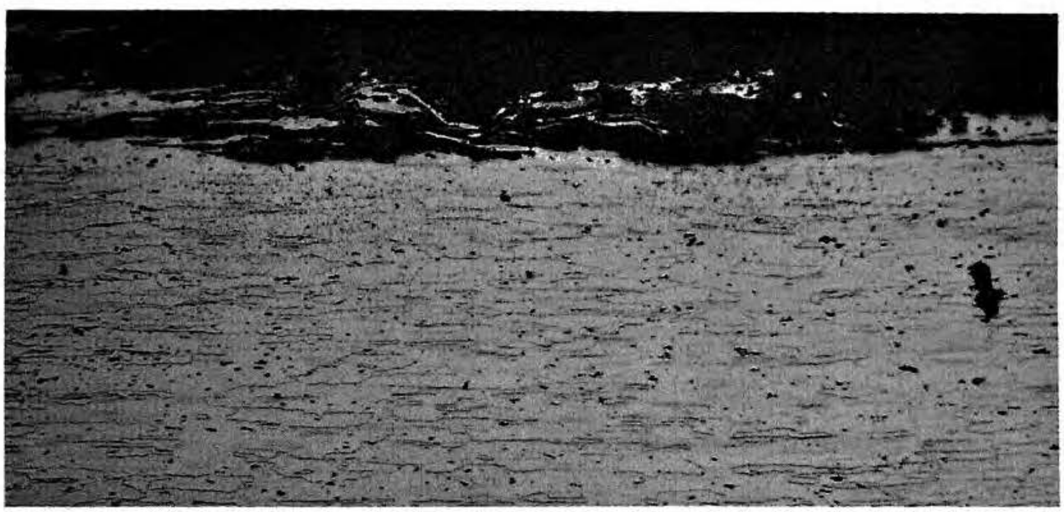
Midplane MAG. 75X

FIGURE 19 – Specimen A4 After Exposure to Acidified Sodium Chloride Corrosion Test



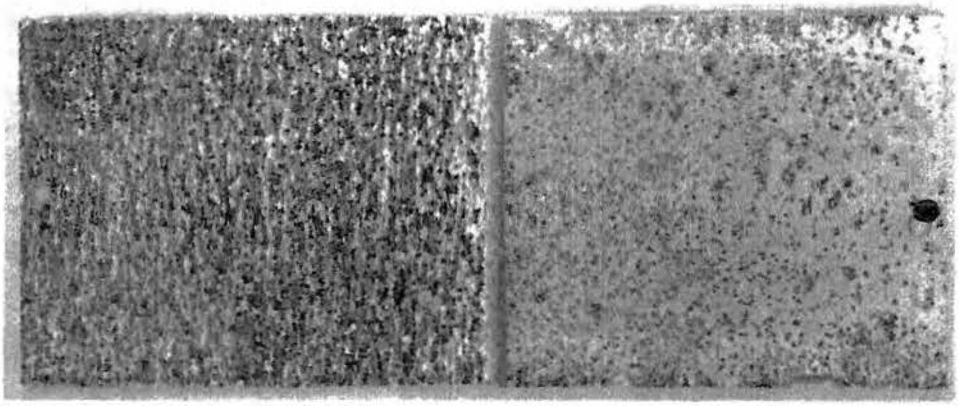
Reduced 50%

Surface MAG. 75X

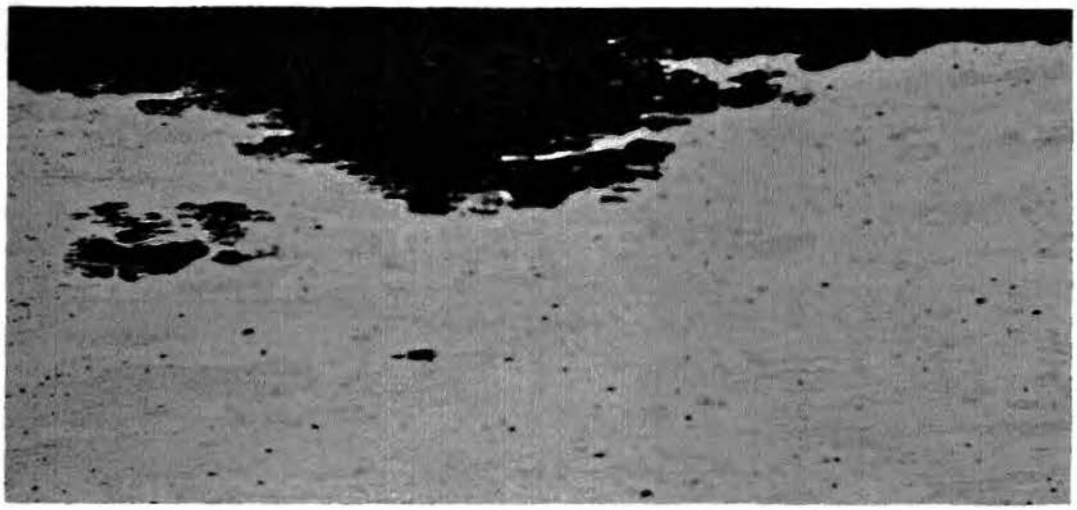


Midplane MAG. 75X

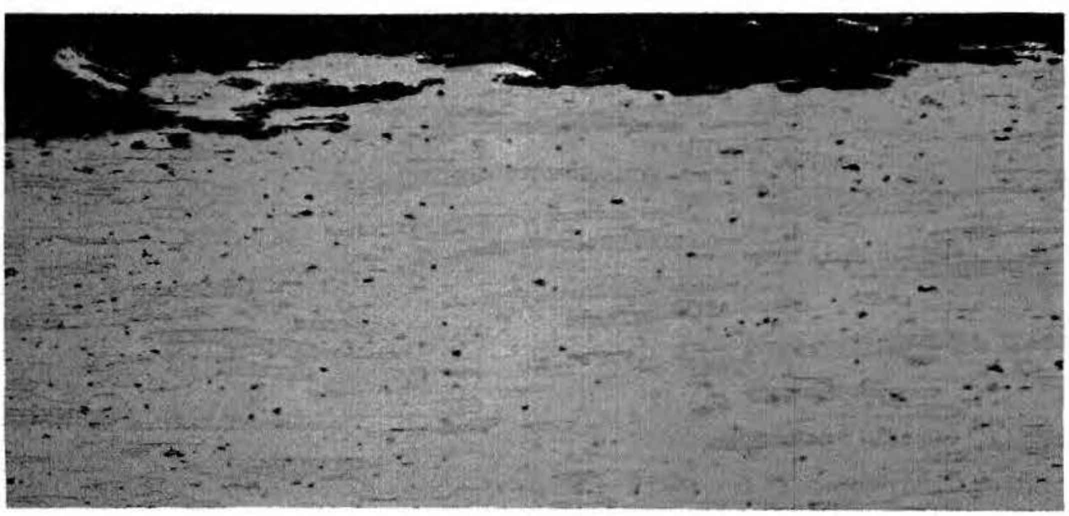
FIGURE 20 – Specimen A4 After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test



Reduced 30%

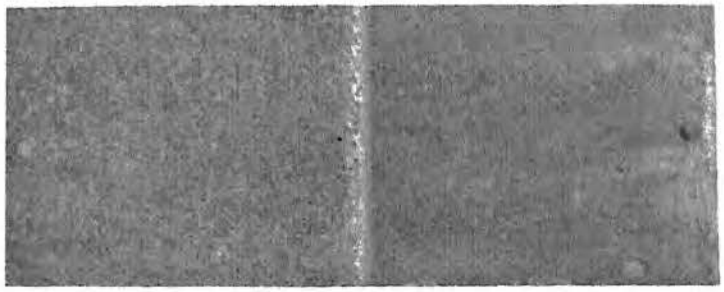


Surface MAG. 75X



Midplane MAG. 75X

FIGURE 21 – Specimen A2 After Exposure to Acidified Sodium Chloride Corrosion Test

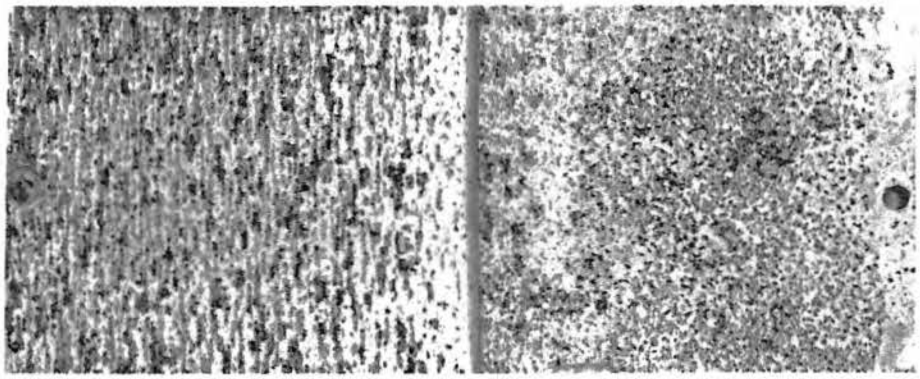


Reduced 50%

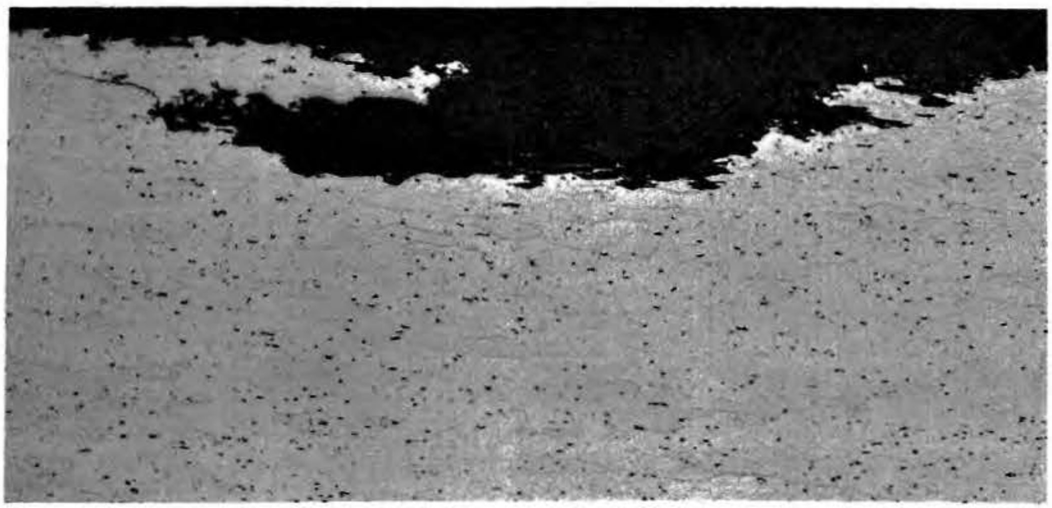
Surface MAG. 75X

Midplane MAG. 75X

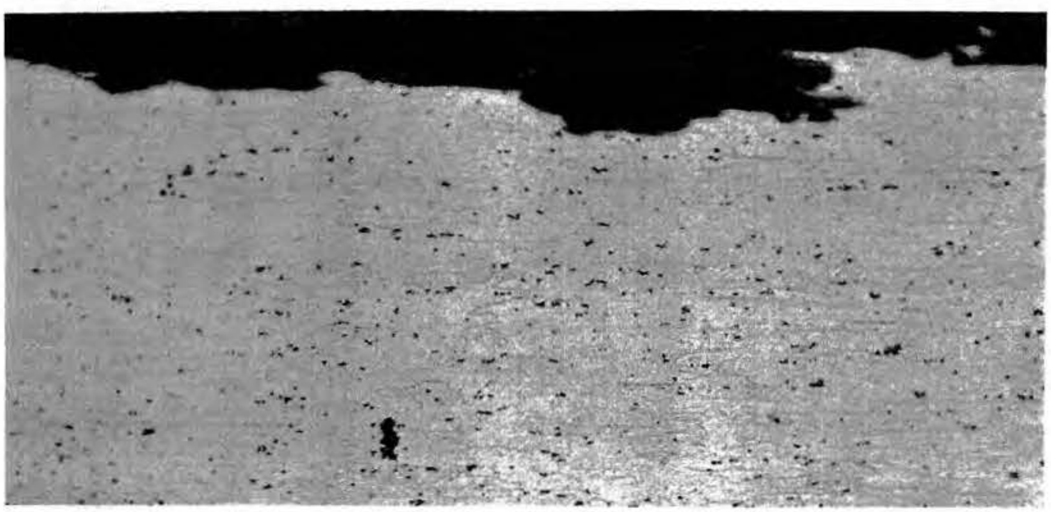
FIGURE 22 – Specimen A2 After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test



Reduced 30%

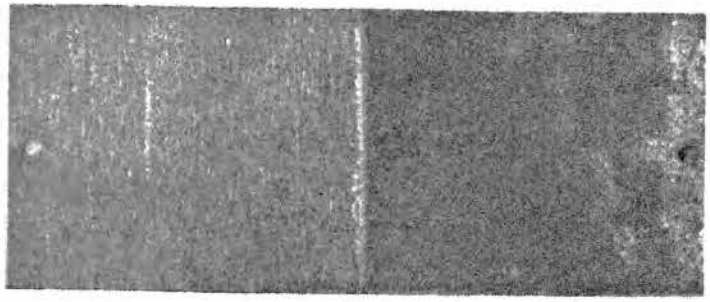


Surface MAG. 75X



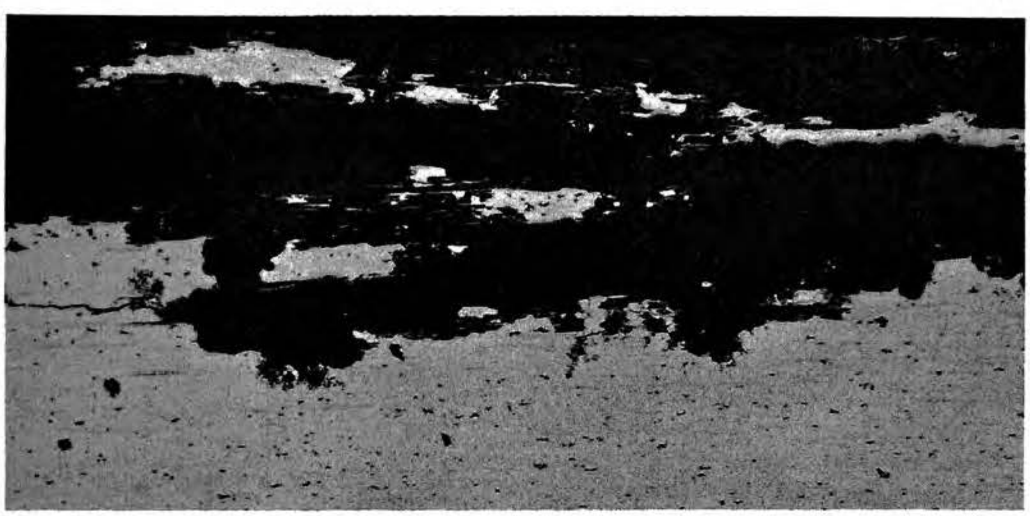
Midplane MAG. 75X

FIGURE 23 – Specimen A3 After Exposure to Acidified Sodium Chloride Corrosion Test



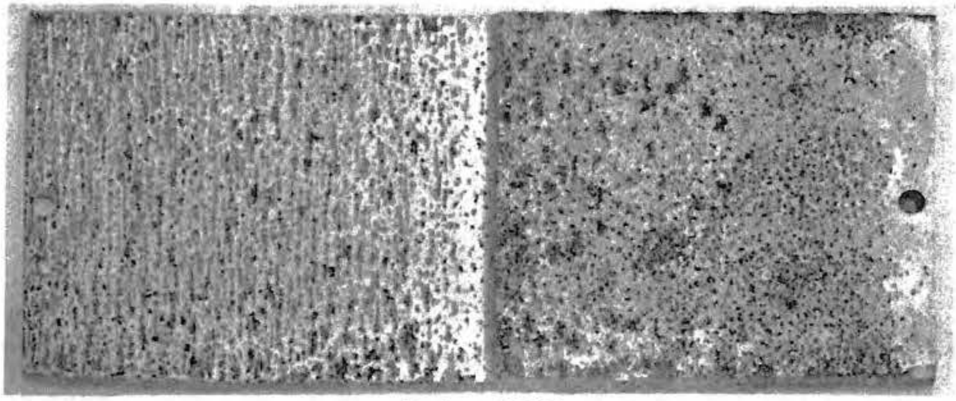
Reduced 50%

Surface MAG. 75X

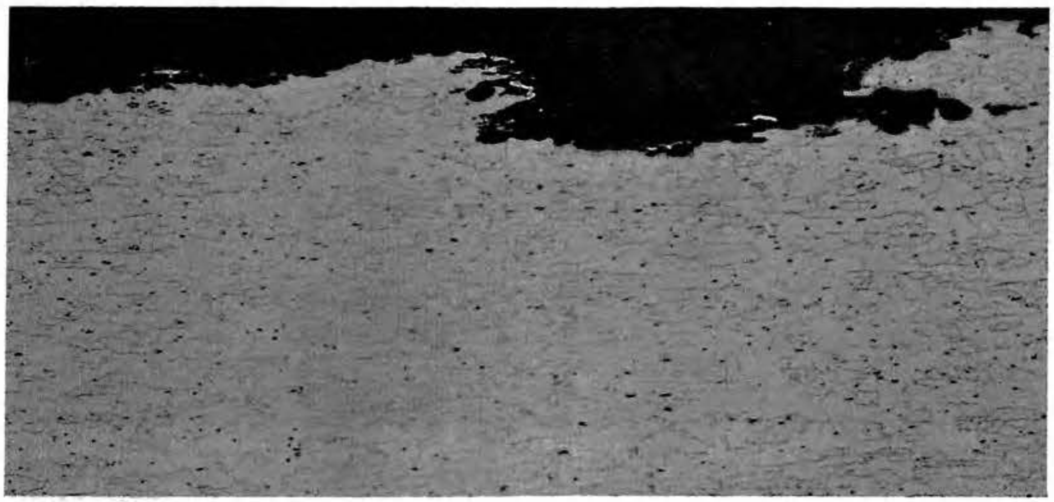


Midplane MAG. 75X

FIGURE 24 – Specimen A3 After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test



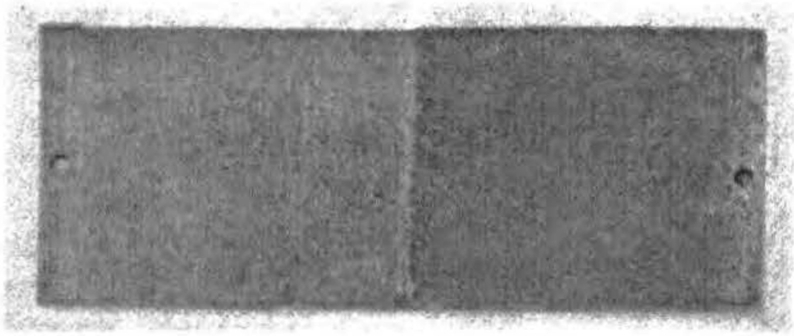
Reduced 30%



Surface MAG. 75X

Midplane MAG. 75X

FIGURE 25 – Specimen A1 After Exposure to Acidified Sodium Chloride Corrosion Test

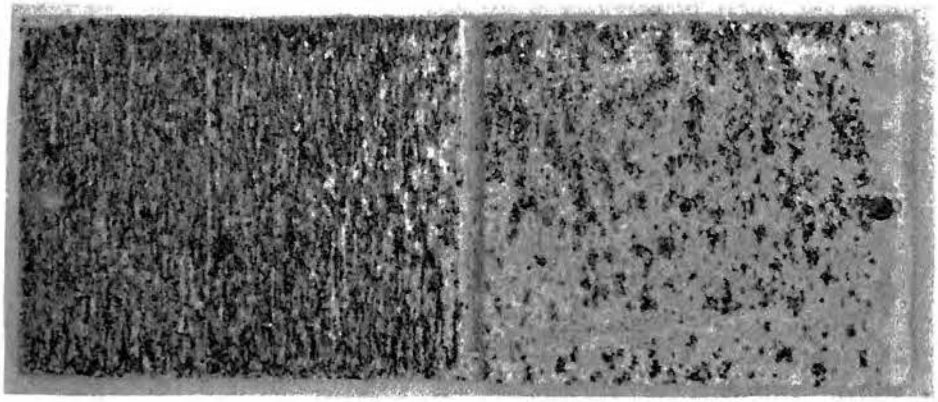


Reduced 50%

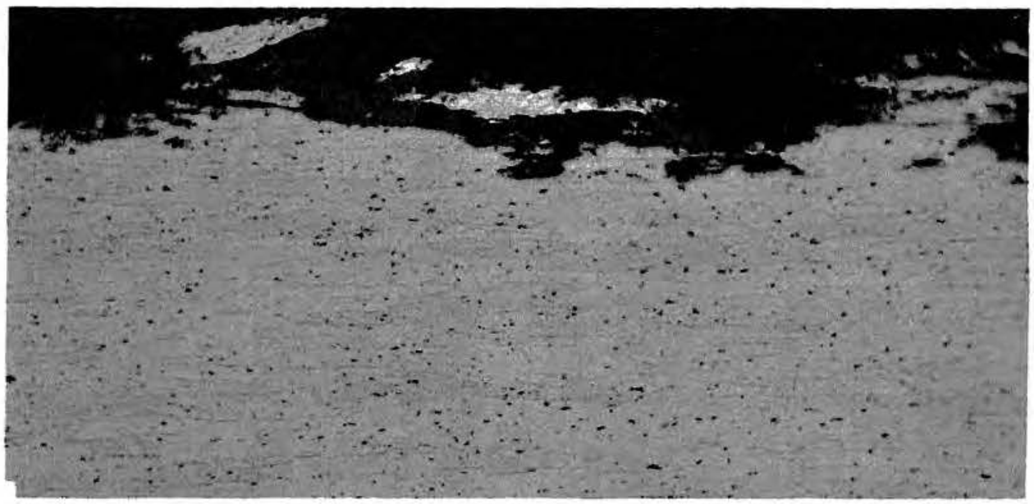
Surface MAG. 75X

Midplane MAG. 75X

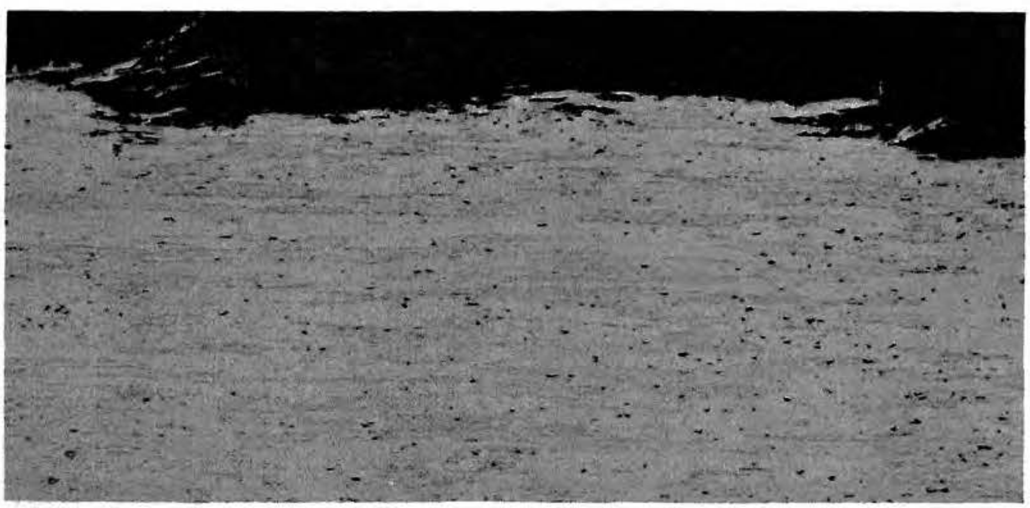
FIGURE 26 – Specimen A1 After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test



Reduced 30%

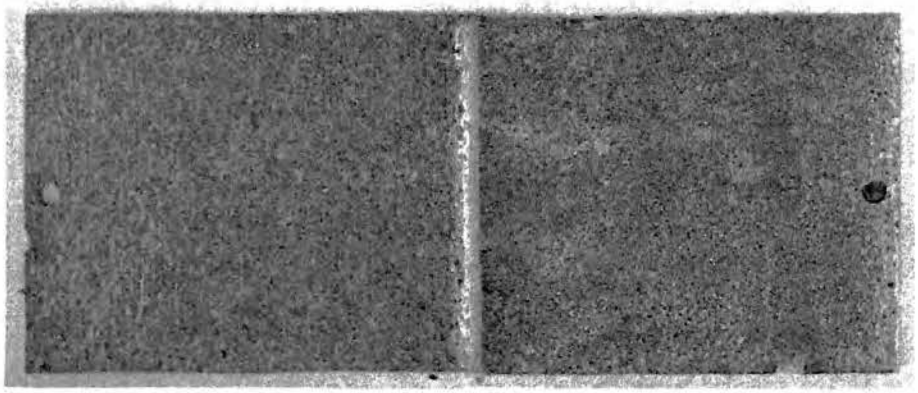


Surface MAG. 75X

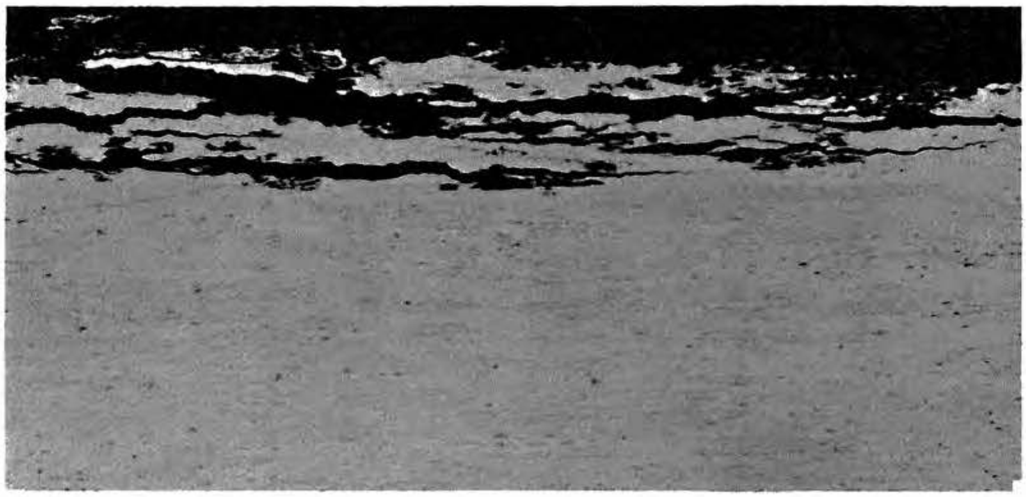


Midplane MAG. 75X

FIGURE 27 – Specimen W1 After Exposure to Acidified Sodium Chloride Corrosion Test



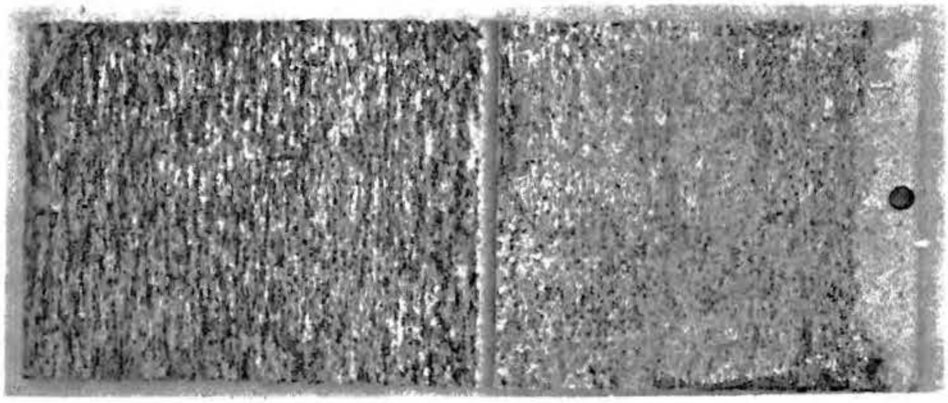
Reduced 50%



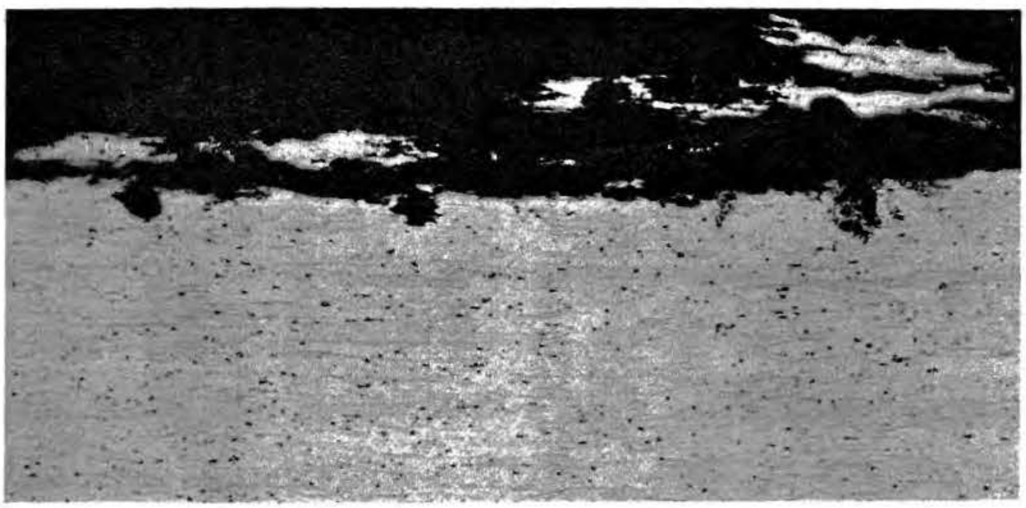
Surface MAG. 75X

Midplane MAG. 75X

FIGURE 28 – Specimen W1 After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test



Reduced 30%



Surface MAG. 75X

Midplane MAG. 75X

FIGURE 29 – Specimen W3 After Exposure to Acidified Sodium Chloride Corrosion Test

specimen reduced 10% and 25% in the annealed condition did in the more severe NaCl plus SO_2 gas environment. No pitting was evident. Extremely severe attack was caused by the NaCl plus SO_2 gas environment as shown in Figures 28 and 30.

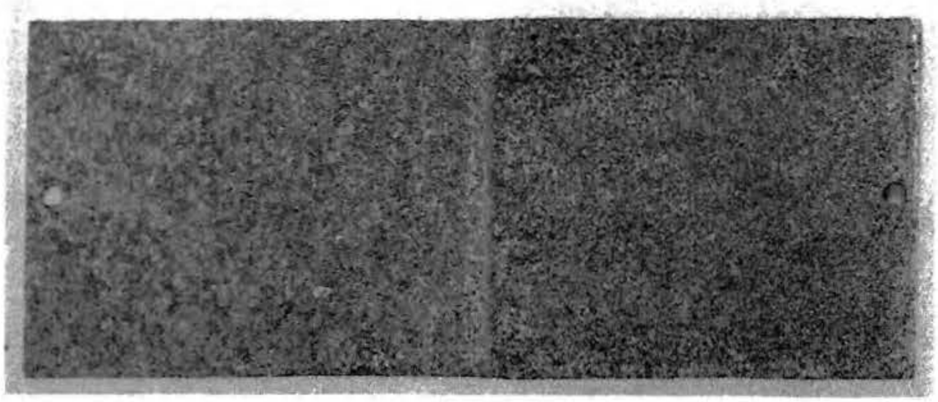
As the reduction in the solution heat treated condition was increased to 40% severe exfoliation attack occurred in both tests as shown in Figures 31 and 32.

Table IV summarizes the type and severity of corrosive attack in each environment for all the 0.390 inch thick specimens.

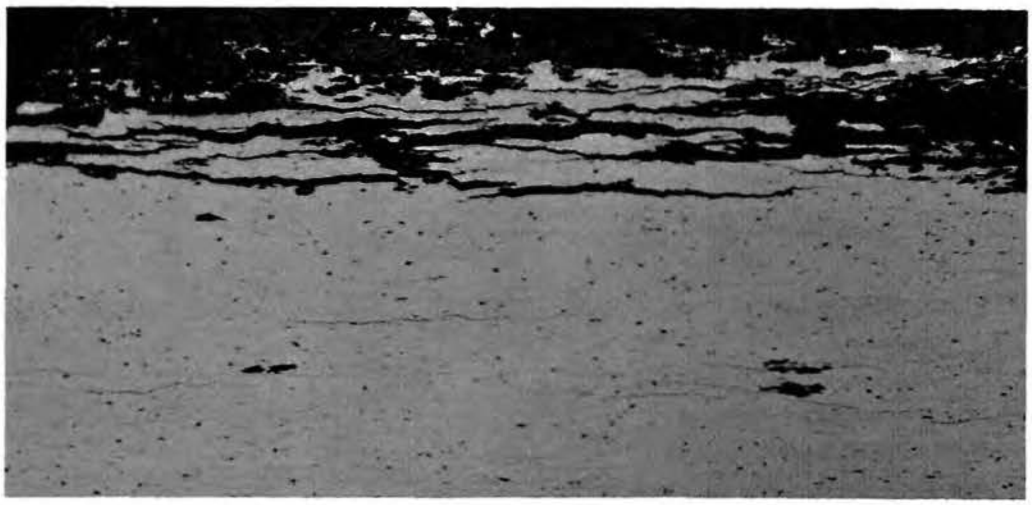
6. Effect of Exfoliation Corrosion Tests on Cold Reduced 0.250 Inch Thick Material

The 0.250 inch thick material control specimen pitted severely in the acidified salt spray environment, Figure 33, but the degree of exfoliation was no more severe than on the 0.390 inch thick material control specimen. However, the exfoliation attack in NaCl plus SO_2 gas was considerably more severe, Figure 34, approaching the most severe exfoliation corrosion evidenced on any of the thicker specimens.

As the amount of reduction increased, the pitting and exfoliation attack in the acidified salt spray lessened, Figures 35, 37 and 39. The severity of exfoliation corrosion caused by the NaCl plus SO_2 gas on the surface of these same specimens was reduced as the amount of cold reduction increased; however, the attack at midplane improved only slightly, Figures 36, 38, and 40. Even though the exfoliation attack at midplane was severe on these specimens which were reduced in the annealed condition, it was not as severe as the attack on the control specimen.



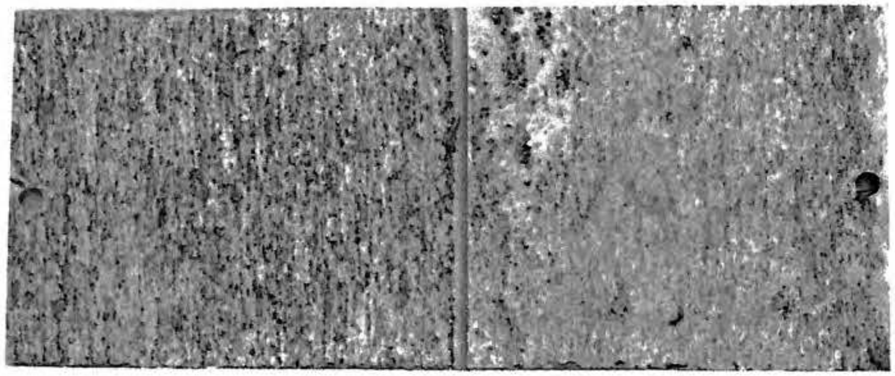
Reduced 50%



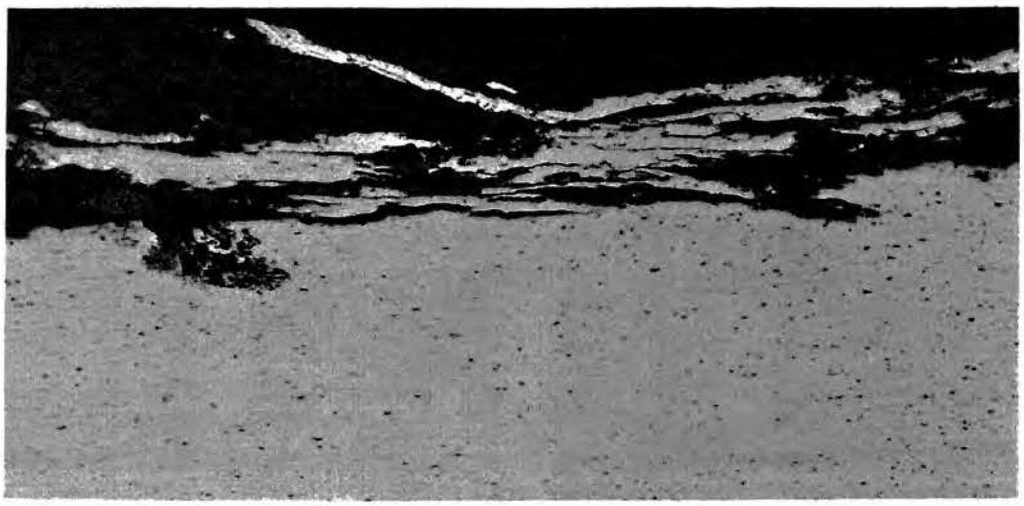
Surface MAG. 75X

Midplane MAG. 75X

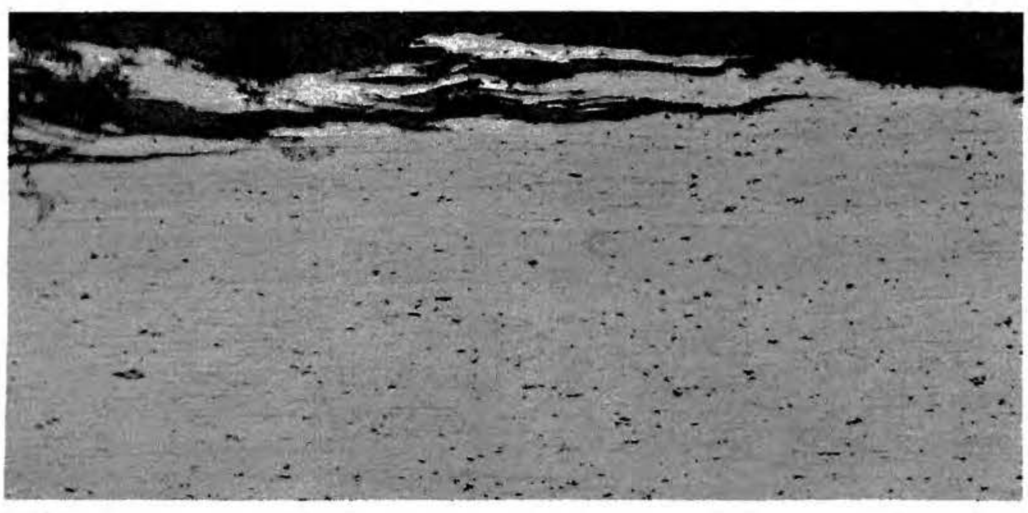
FIGURE 30 – Specimen W3 After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test



Reduced 30%

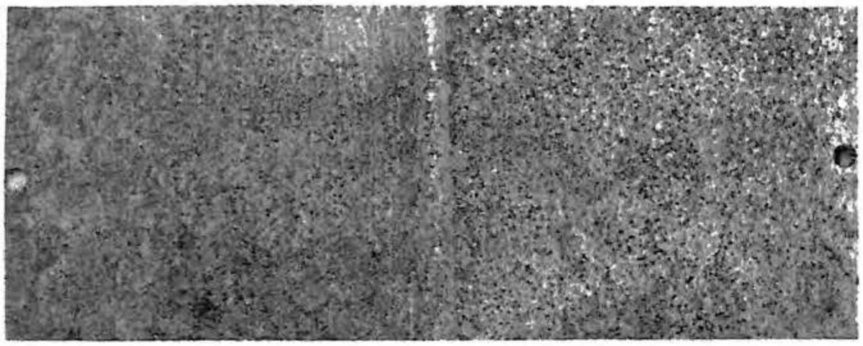


Surface MAG. 75X

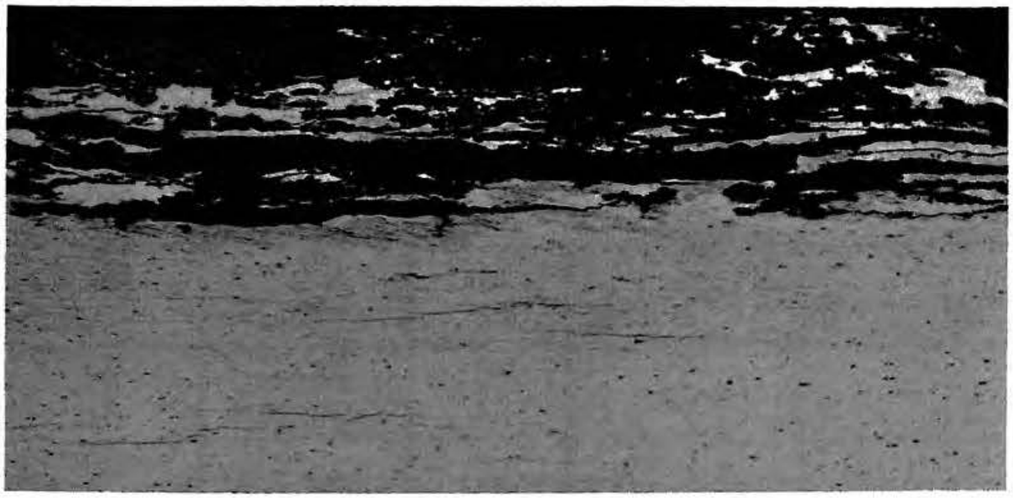


Midplane MAG. 75X

FIGURE 31 – Specimen W4 After Exposure to Acidified Sodium Chloride Corrosion Test



Reduced 50%



Surface MAG. 75X

Midplane MAG. 75X

FIGURE 32 – Specimen W4 After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test

TABLE IV

RESULTS OF EXFOLIATION CORROSION TESTS

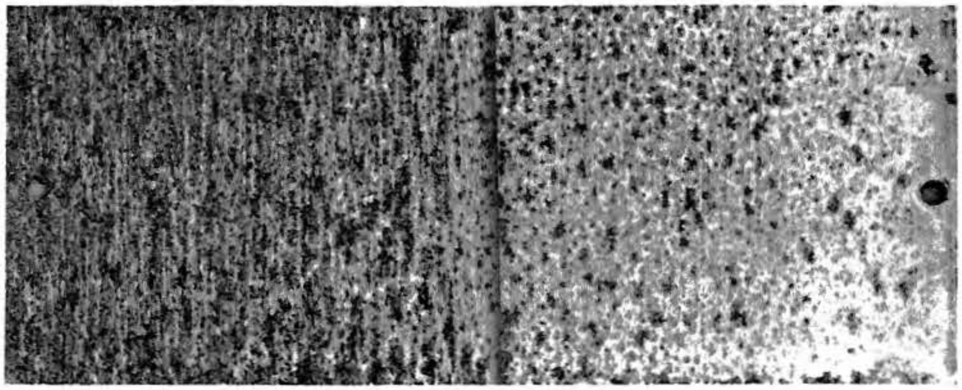
MATERIAL REDUCED FROM 0.390 INCH

pecimen No.	Reduction (%)	Location	Environment		
			Acidified Salt		Salt Spray + SO_2
			Pitting	Exfoliation	Exfoliation
A4	0	Surface	2	1	2
		Midplane	1	1	3
A2	10	Surface	3	1	4
		Midplane	1	0	5
A3	25	Surface	3	1	4
		Midplane	1	1	4
A1	40	Surface	3	0	1
		Midplane	5	0	3
W1	10	Surface	-	2	8
		Midplane	-	1	7
W3	25	Surface	-	3	8
		Midplane	-	4	7
W4	40	Surface	-	7	8
		Midplane	-	6	8
A5	0	Surface	1	-	-
		Midplane	1	-	1

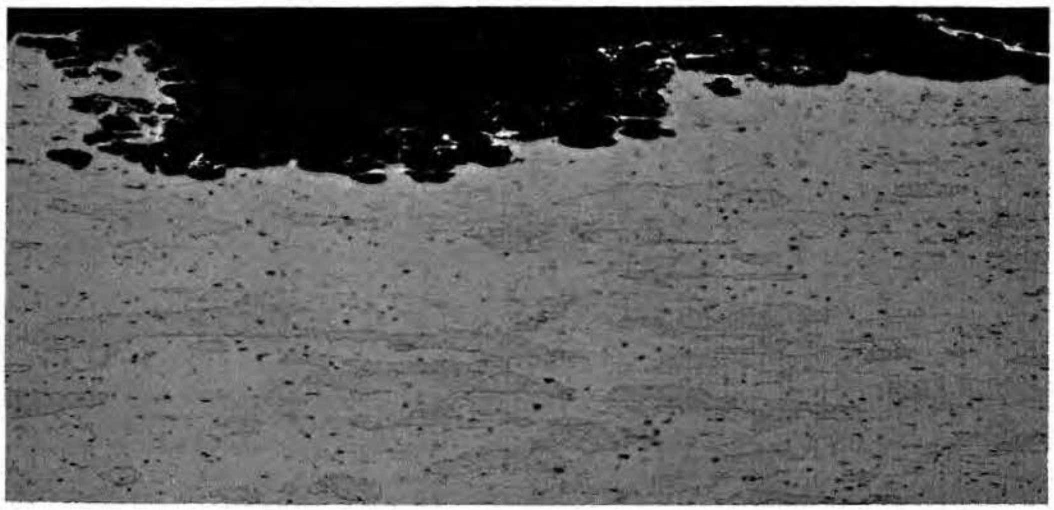
Exfoliation and Pitting Rating

1 = Minor

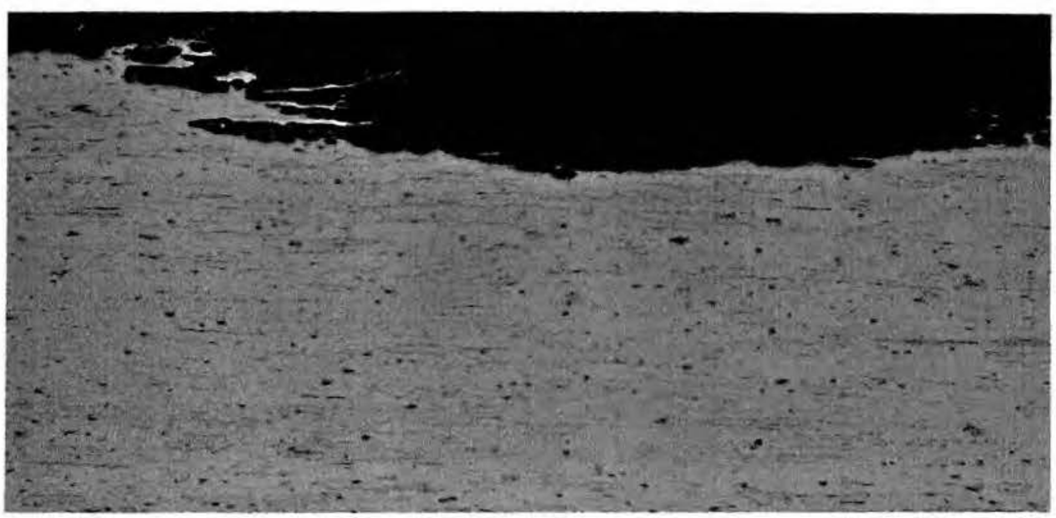
10 = Most Severe



Reduced 30%

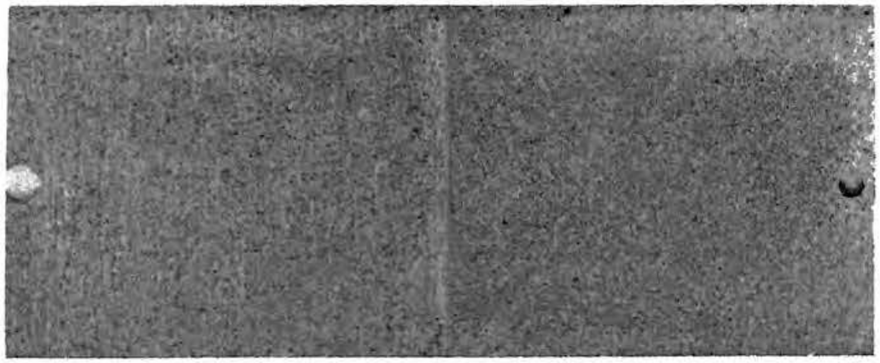


Surface MAG. 75X



Midplane MAG. 75X

FIGURE 33 – Specimen W4X After Exposure to Acidified Sodium Chloride Corrosion Test



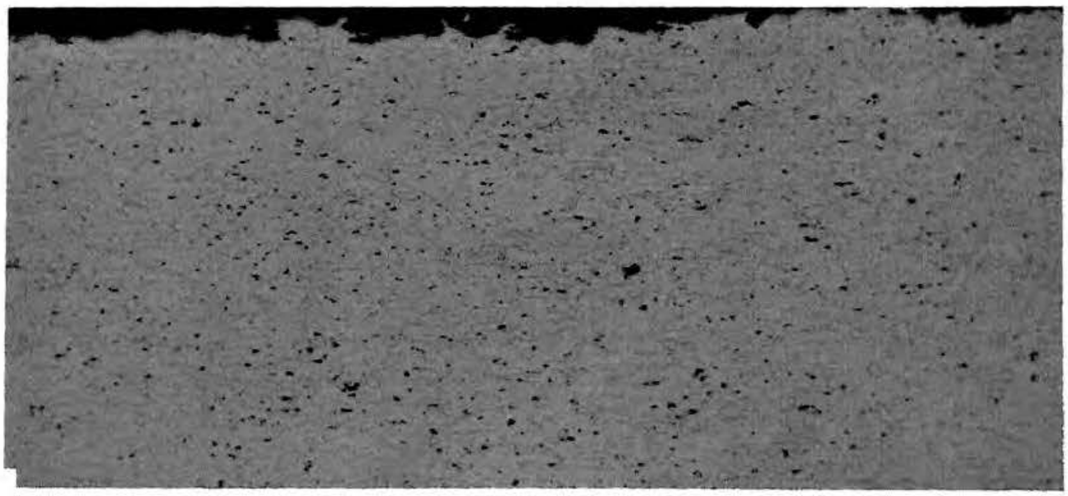
Reduced 50%

Surface MAG. 75X

Midplane MAG. 75X

FIGURE 34 – Specimen W4X After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test

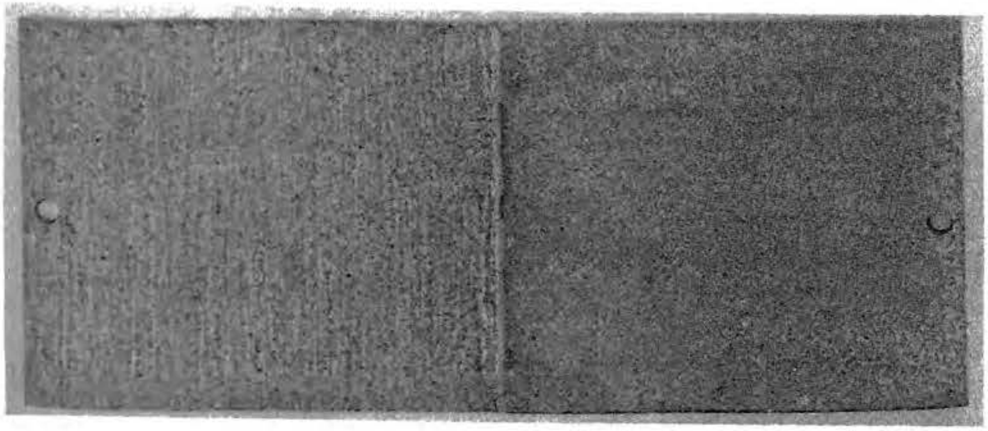
Reduced 30%



Surface MAG. 75X

Midplane MAG. 75X

FIGURE 35 – Specimen A4X After Exposure to Acidified Sodium Chloride Corrosion Test

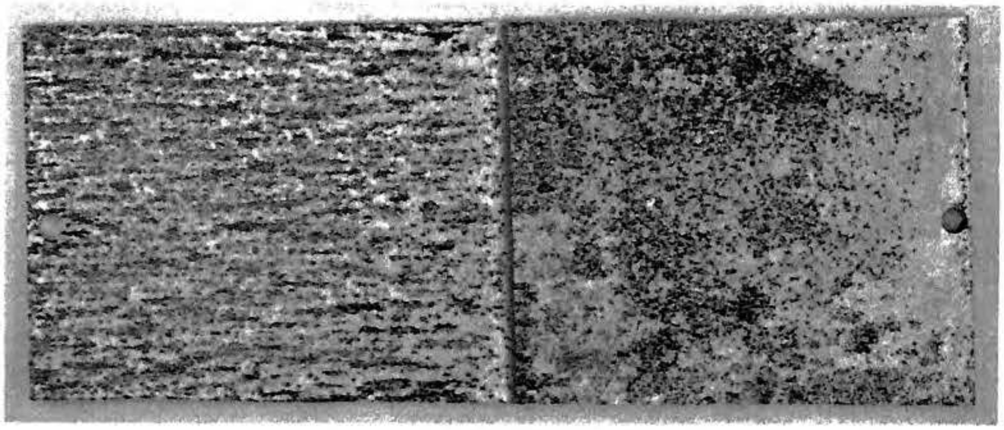


Reduced 30%

Surface MAG. 75X

Midplane MAG. 75X

FIGURE 36 – Specimen A4X After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test

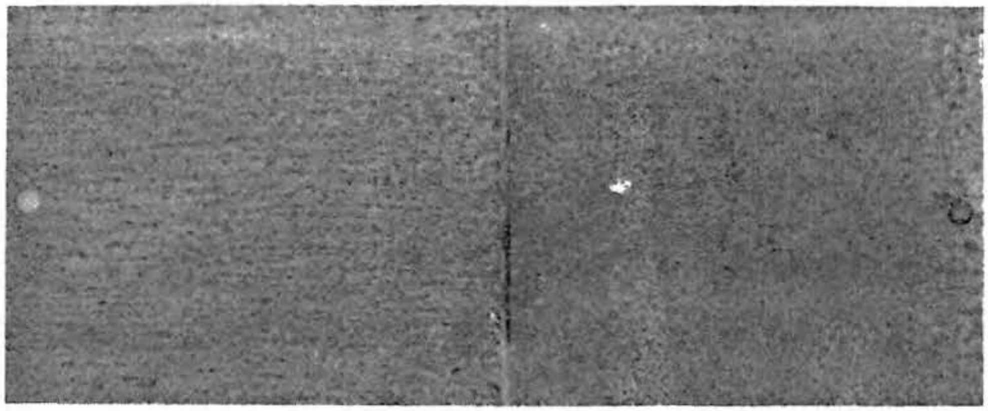


Reduced 30%

Surface MAG. 75X

Midplane MAG. 75X

FIGURE 37 – Specimen A3X After Exposure to Acidified Sodium Chloride Corrosion Test

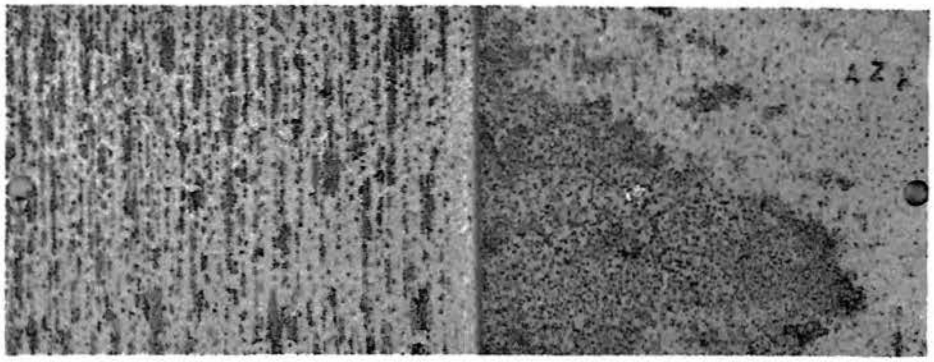


Reduced 30%

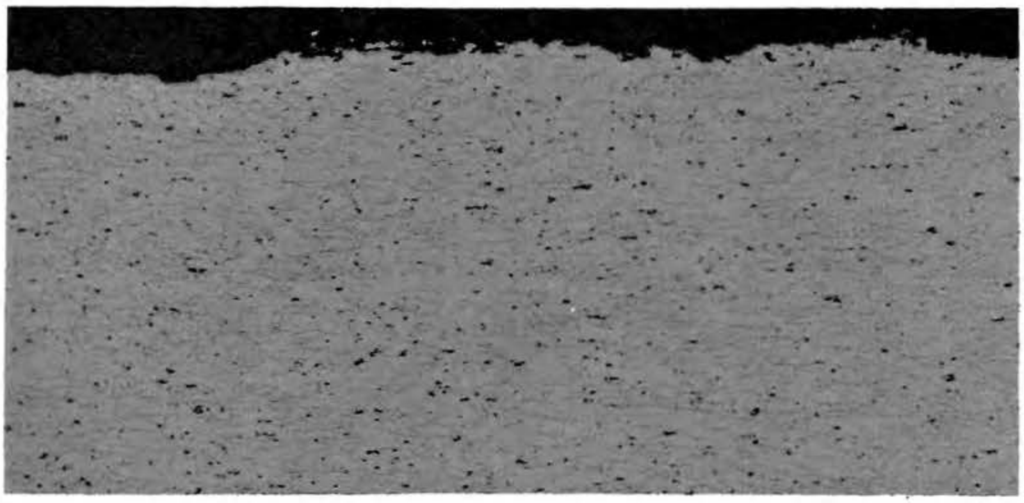
Surface MAG. 75X

Midplane MAG. 75X

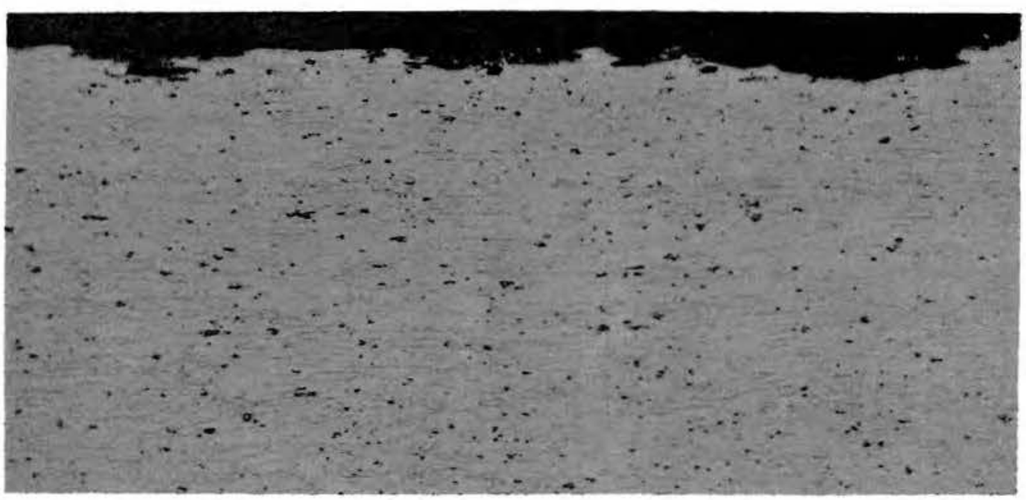
FIGURE 38 – Specimen A3X After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test



Reduced 30%

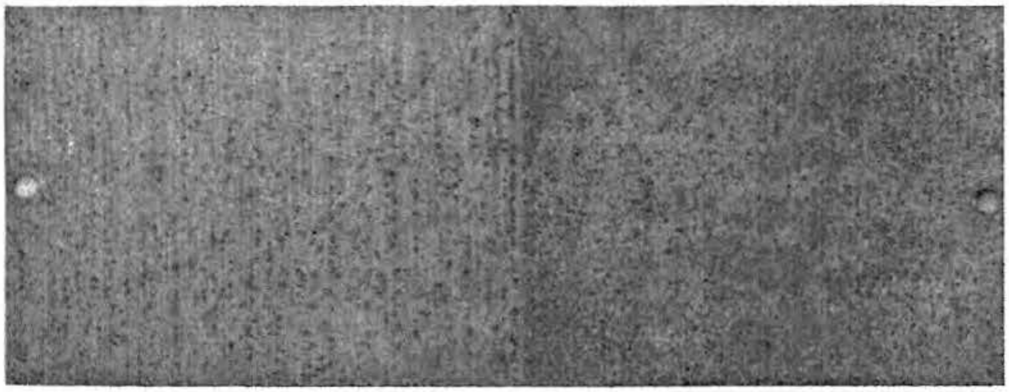


Surface MAG. 75X

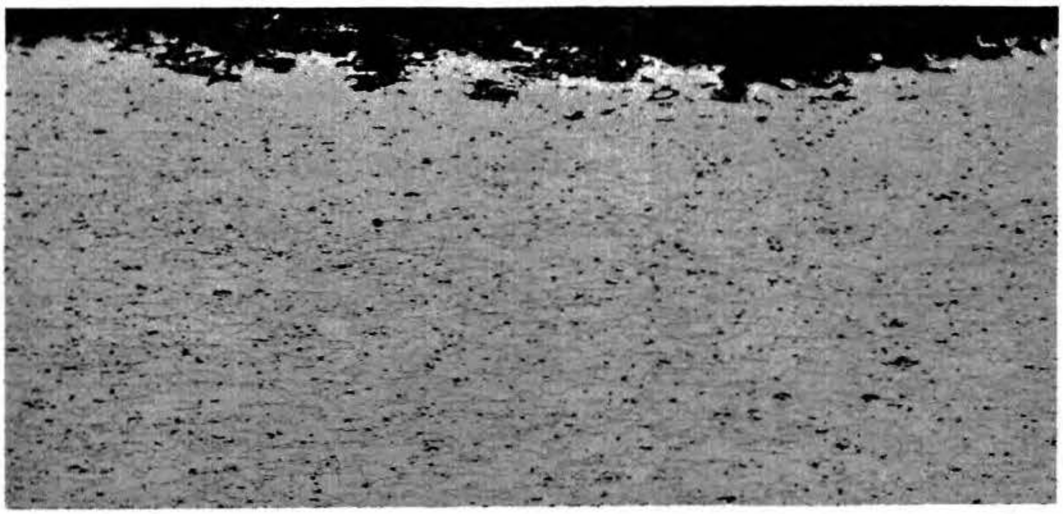


Midplane MAG. 75X

FIGURE 39 – Specimen A2X After Exposure to Acidified Sodium Chloride Corrosion Test



Reduced 30%



Surface MAG. 75X

Midplane MAG. 75X

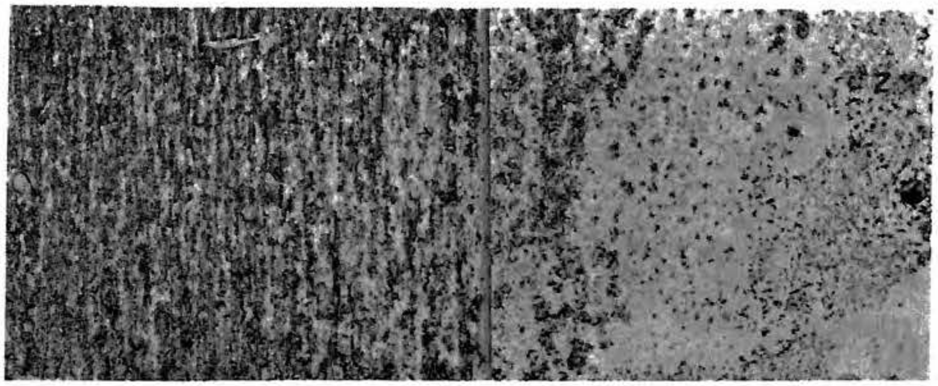
FIGURE 40 – Specimen A2X After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test

The 0.250 inch thick material reduced in the solution heat treated condition 10% and 25% was pitted less but exfoliated more severely, Figures 41 and 43, in the acidified salt spray than the unreduced control specimen. The exfoliation corrosion attack on these specimens in the NaCl plus SO_2 gas test increased dramatically, Figures 42 and 44, exceeding the most severe exfoliation attack found in any of the thicker specimens. The 0.250 inch specimen reduced 40% in the solution heat treated condition exhibited very severe exfoliation in the acidified salt, Figure 45, and the most severe exfoliation found on any specimen in the NaCl plus SO_2 gas environment, Figure 46.

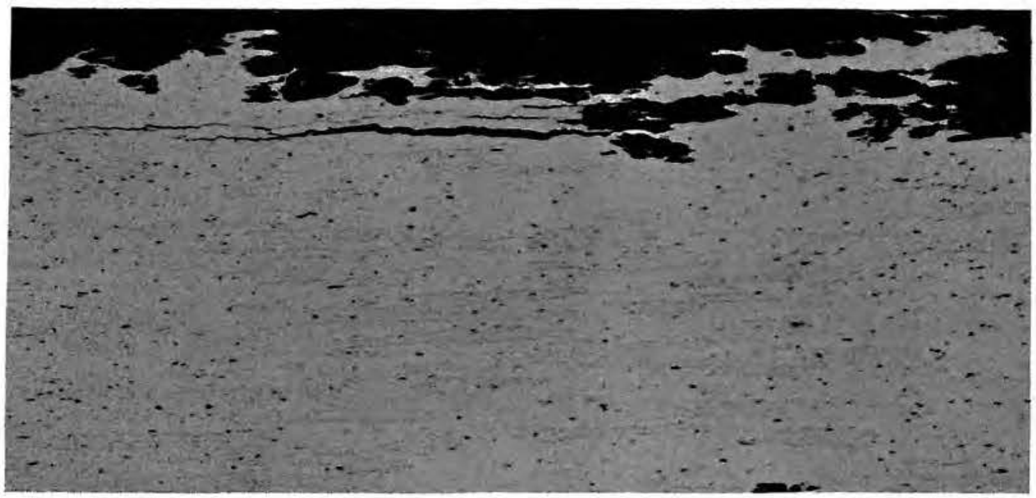
Table V summarizes the type and severity of corrosive attack caused by each environment for all the 0.250 inch thick specimens.

7. Additional Effects of Exfoliation Corrosion Tests

In addition to pitting and surface and midplane exfoliation caused by both corrosive environments, some specimens exfoliated severely enough at midplane to be classified at "midplane cracking". A summary of the specimens which exhibited midplane cracking is presented in Table VI. All of the 0.390 inch thick material reduced in the solution heat treated condition, "cracked" in both the acidified salt and in the NaCl plus SO_2 gas environments. The cold reduced 0.250 inch thick material, reduced in the solution heat treated condition, showed only minor to moderate "midplane cracking" in the NaCl plus SO_2 gas tests. The "midplane cracking" is severe exfoliation attack at the radius of the specimens and demonstrates the reduction in structural integrity which can result from this type attack. Examples of the type of "midplane cracking" experienced are shown in Figure 47.



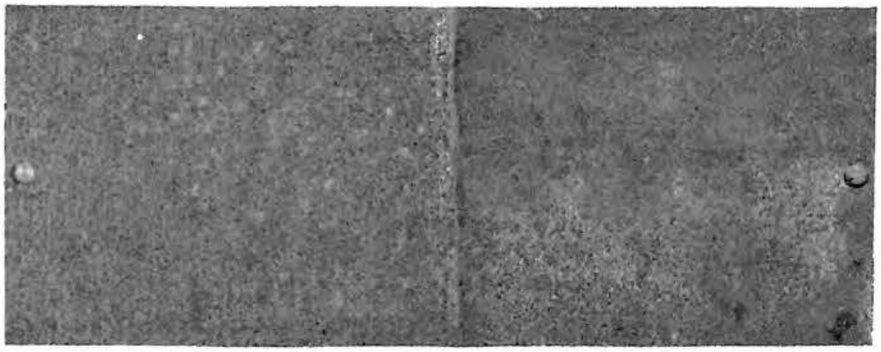
Reduced 30%



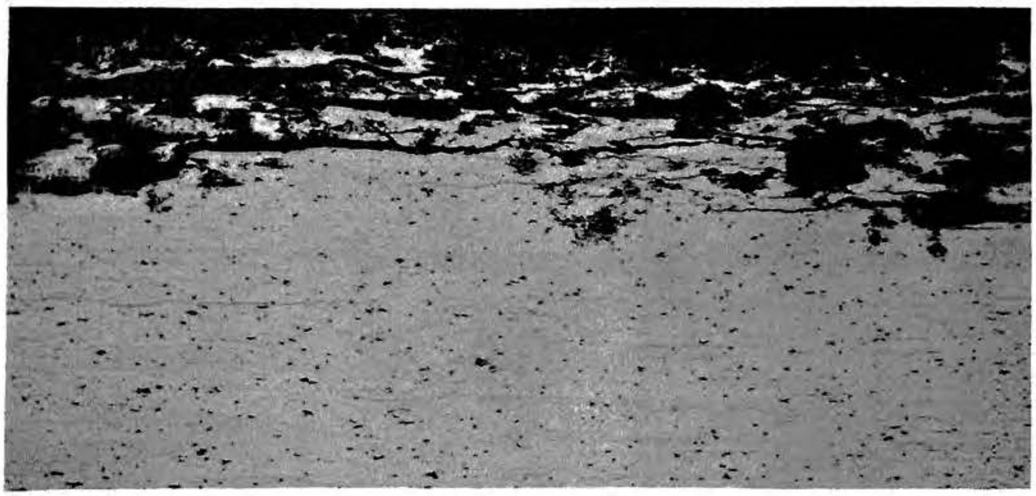
Surface MAG. 75X

Midplane MAG. 75X

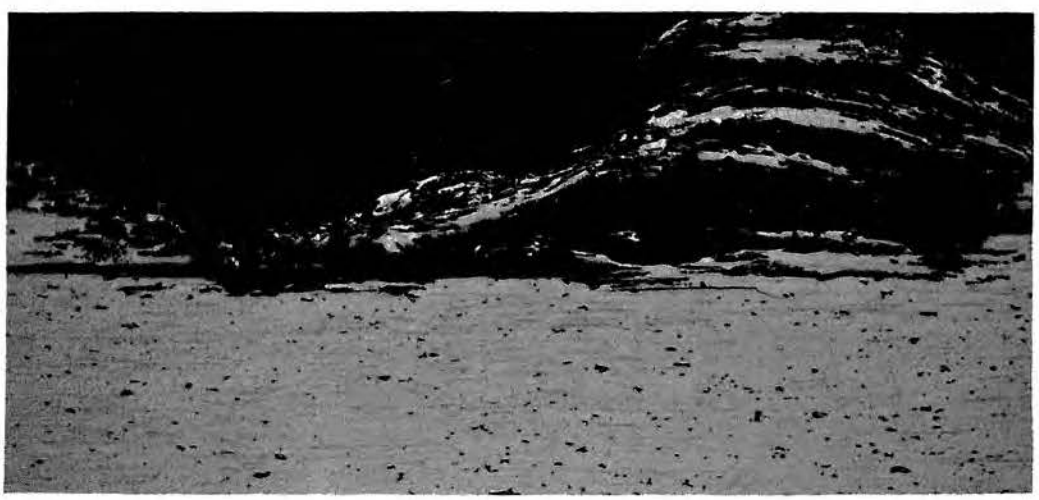
FIGURE 41 – Specimen W2X After Exposure to Acidified Sodium Chloride Corrosion Test



Reduced 50%

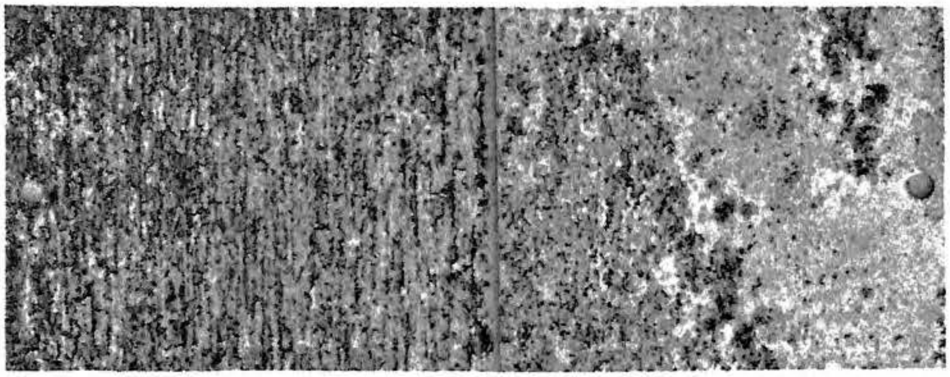


Surface MAG. 75X

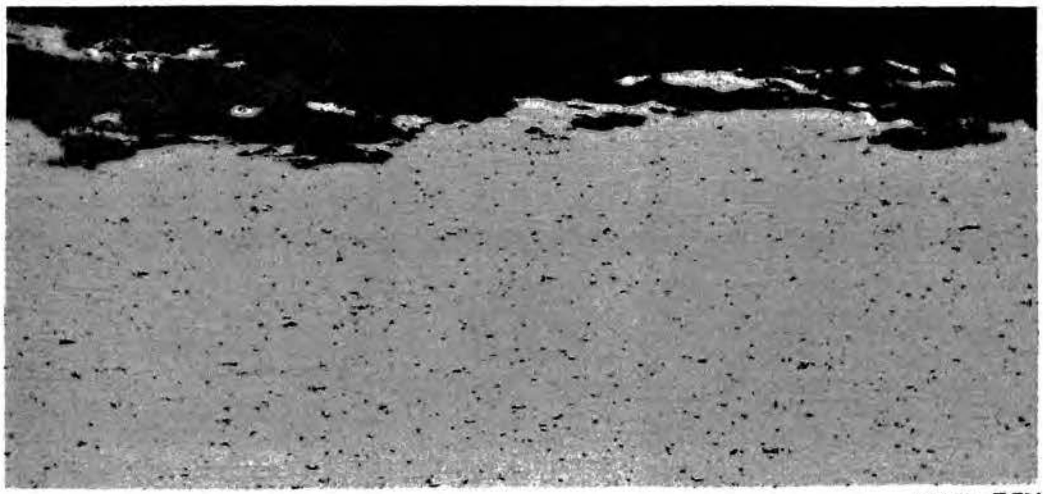


Midplane MAG. 75X

FIGURE 42 – Specimen W2X After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test



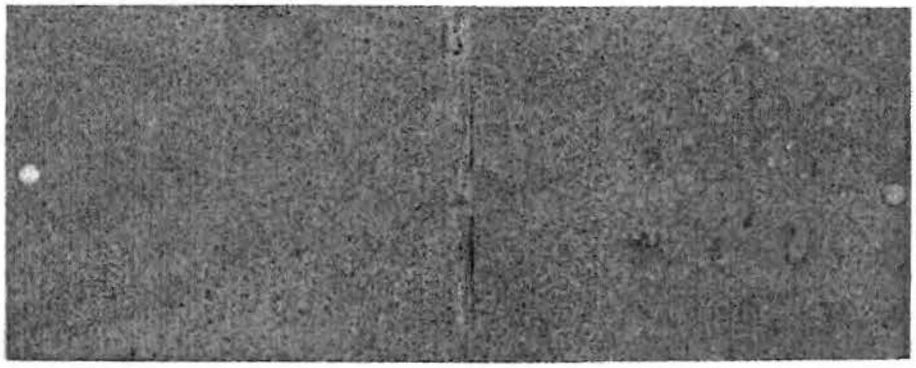
Reduced 30%



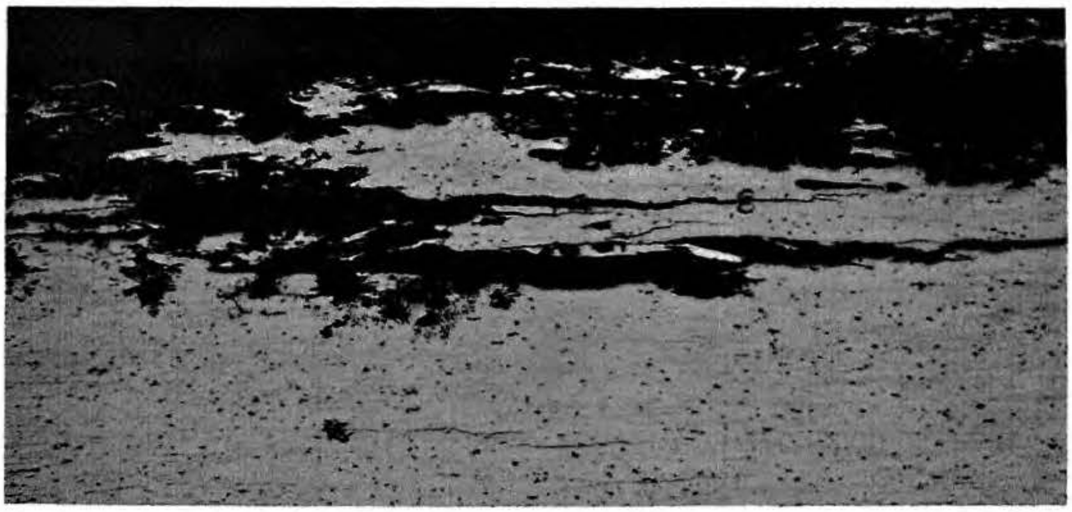
Surface MAG. 75X

Midplane MAG. 75X

FIGURE 43 – Specimen W3X After Exposure to Acidified Sodium Chloride Corrosion Test



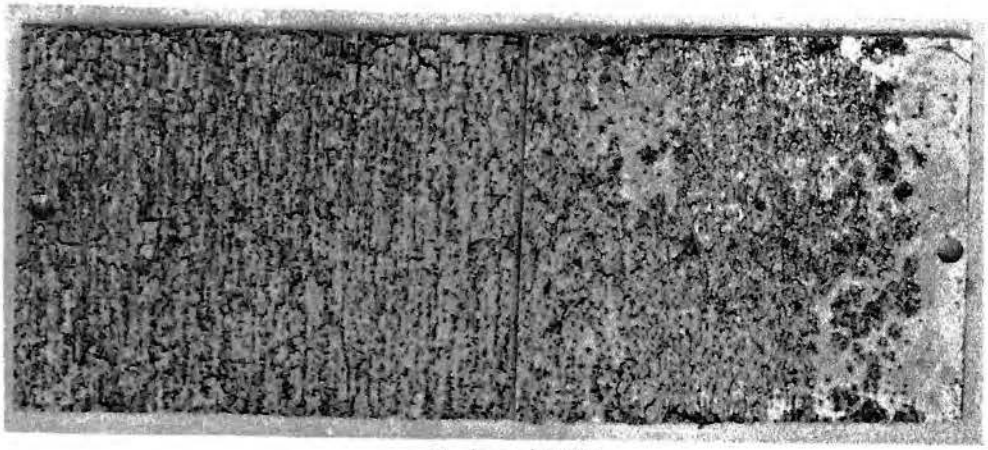
Reduced 50%



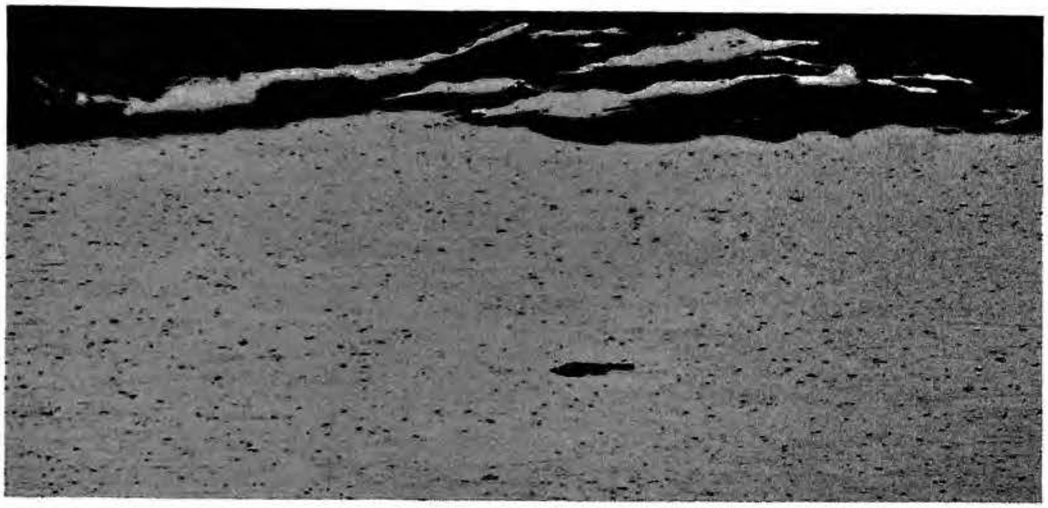
Surface MAG. 75X

Midplane MAG. 75X

FIGURE 44 – Specimen W3X After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test



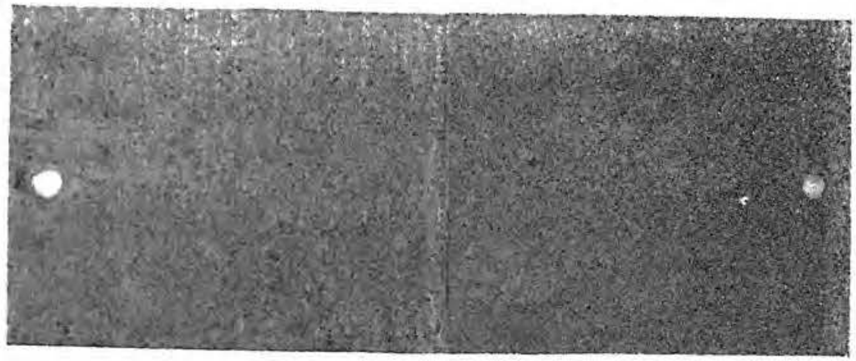
Reduced 30%



Surface MAG. 75X

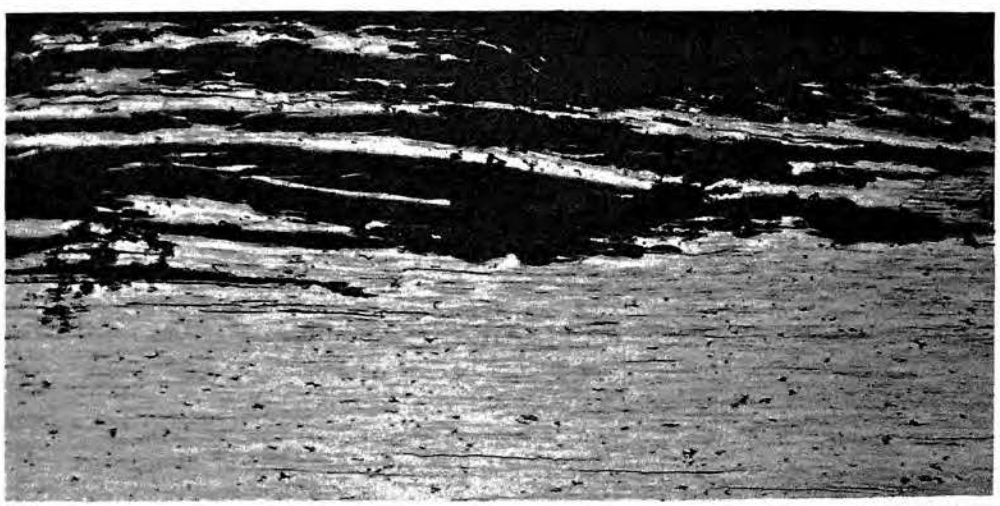
Midplane MAG. 75X

FIGURE 45 – Specimen W1X After Exposure to Acidified Sodium Chloride Corrosion Test



Reduced 50%

Surface MAG. 75X



Midplane MAG. 75X

IGURE 46 – Specimen W1X After Exposure to Sodium Chloride Plus Sulfur Dioxide Gas Corrosion Test

TABLE V

RESULTS OF EXFOLIATION CORROSION TESTS

MATERIAL REDUCED FROM 0.250 INCH

Specimen No.	Reduction (%)	Location	Environment		
			Acidified Salt		Salt Spray + SO_2
			Pitting	Exfoliation	Exfoliation
W4X	0	Surface	5	1	5
		Midplane	3	1	7
A4X	10	Surface	2	1	3
		Midplane	1	2	7
A3X	25	Surface	1	1	3
		Midplane	0	1	6
A2X	40	Surface	1	0	1
		Midplane	1	0	5
W2X	10	Surface	1	3	8
		Midplane	2	2	8
W3X	25	Surface	1	2	9
		Midplane	1	3	9
W1X	40	Surface	-	6	10
		Midplane	-	7	10

Exfoliation and Pitting Rating

1 = Minor

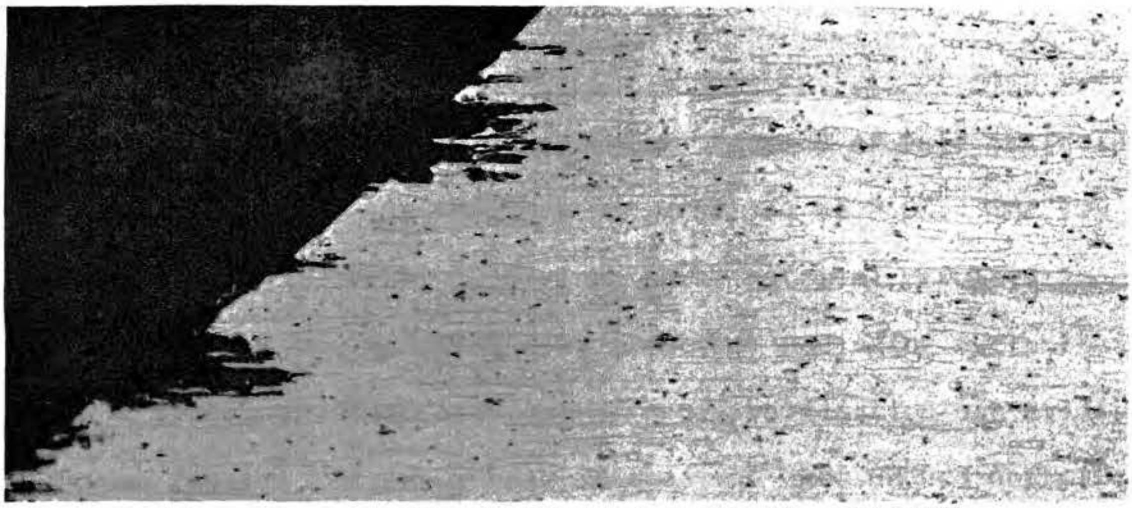
↓

10 = Most Severe

TABLE VI

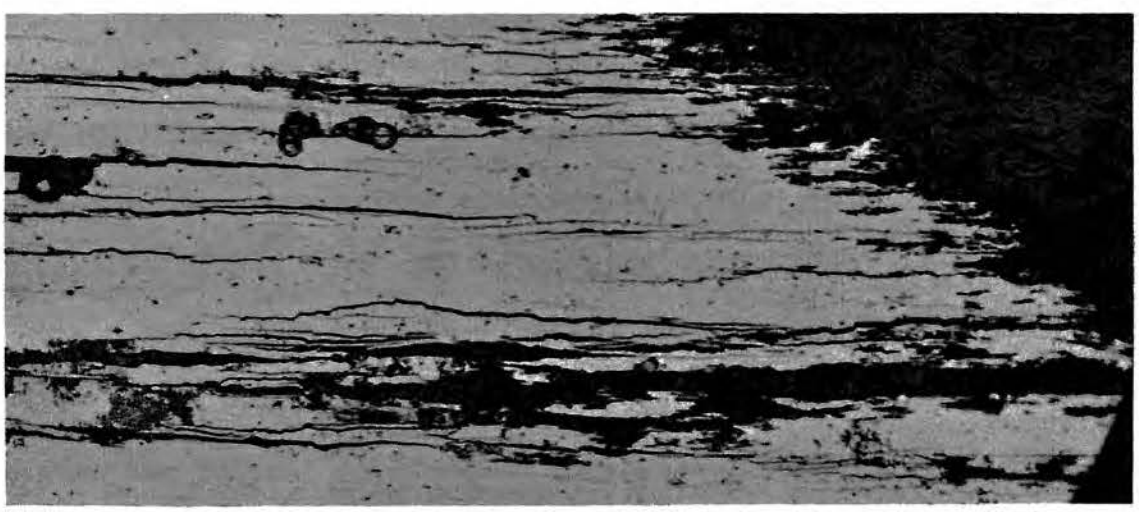
MIDPLANE CRACKING RESULTS

Specimen No.	Reduction (%)	Environment	
		Acidified NaCl	NaCl + SO_2
A4	0	None	None
A2	10		
A3	25		
A1	40		
W4X	0		
A4X	10		
A3X	25		
A2X	40		
W1	10	Severe	Very Severe
W3	25		
W4	40		
W2X	10	None	Minor
W3X	25	Minor	Moderate
W1X	40	None	None
A5	0	None	None



None – Specimen A4 – Acidified Sodium Chloride – MAG.75X

Moderate – Specimen W3X – Sodium Chloride Plus Sulfur Dioxide Gas – MAG. 75X



Very Severe – Specimen W3 – Sodium Chloride Plus Sulfur Dioxide Gas – MAG. 75X

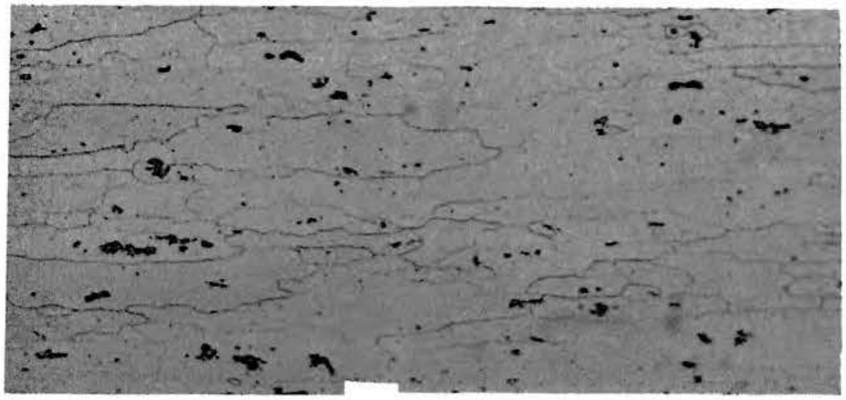
FIGURE 47 – Examples of Midplane Cracking

8. <u>Effect of Cold Reduction on the Microstructure of Each Specimen</u>

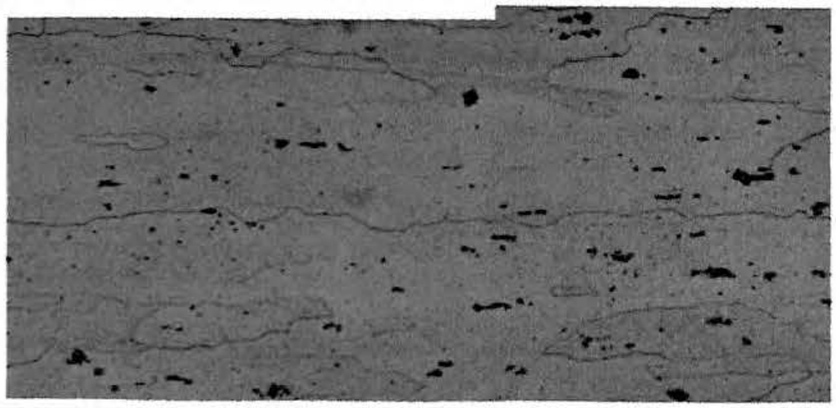
To determine the effect of the various amounts of cold reduction n the microstructure, each specimen was examined at 250X. The 10% eduction given the annealed 0.390 inch thick material resulted in arger grains after the solution heat treatment and aging cycle than he control specimen. Some evidence of recrystallization was evident. The specimen reduced 25%, then heat treated and aged showed definite recrystallization which resulted in a smaller grain size than the control specimen. The specimen reduced 40% was recrystallized and produced a smaller, much less directional grain structure. The specimen showing the greatest amount of recrystallization also had the most resistance to exfoliation corrosion attack. A comparison of these microstructures are shown in Figure 48.

The 0.390 inch thick specimens reduced 10%, 25% and 40% in the solution heat treated condition and only aged at 250°F after cold reduction showed a progressively more directional grain structure as the amount of cold reduction increased. These specimens also exhibited grains indicative of a highly worked structure as shown in Figure 49. These specimens were considerably more susceptible to exfoliation corrosion attack than the specimens which were solution heat treated, 870°F, and aged after cold reduction.

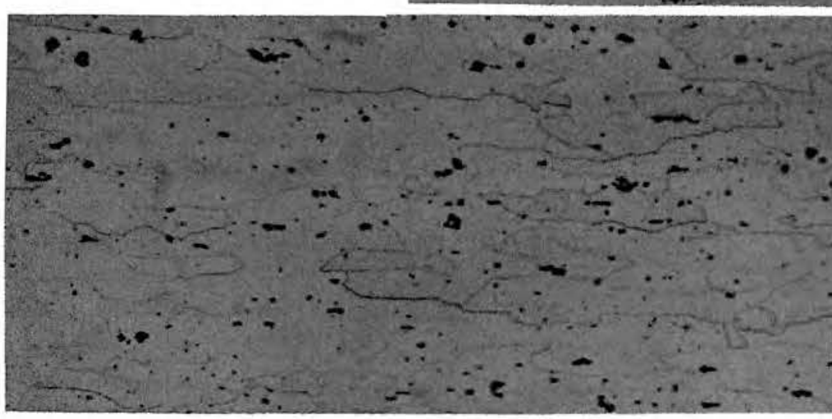
The 0.250 inch thick material reduced 10% and then solution heat treated, 870°F, and aged, had a larger, less directional grain structure than the control specimen. The specimen reduced 25% showed definite signs of recrystallization. The specimen reduced 40% demonstrated a large degree of recrystallization exhibiting a very uniform, non-directional grain structure. This specimen's resistance to exfoliation



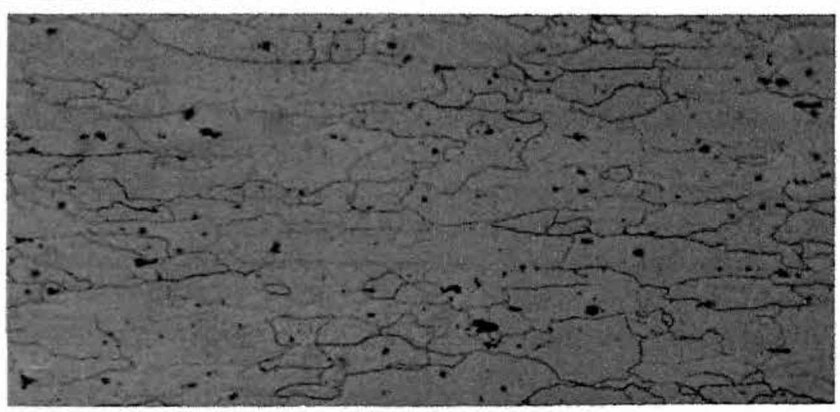
Specimen A4
Control
MAG. 250X



Specimen A2
10% Reduction
MAG. 250X

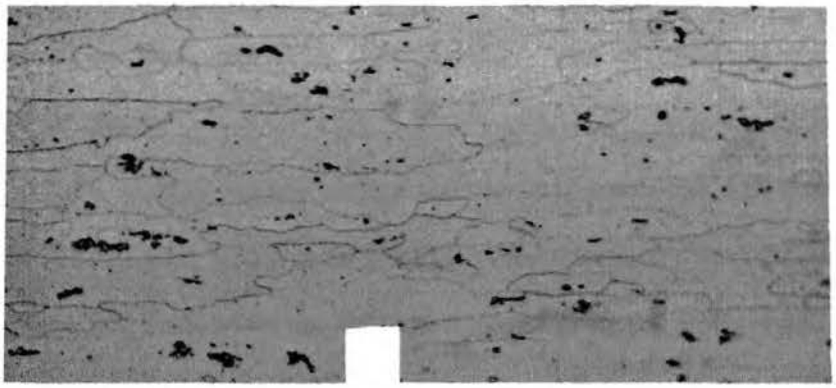


Specimen A3
25% Reduction
MAG. 250X



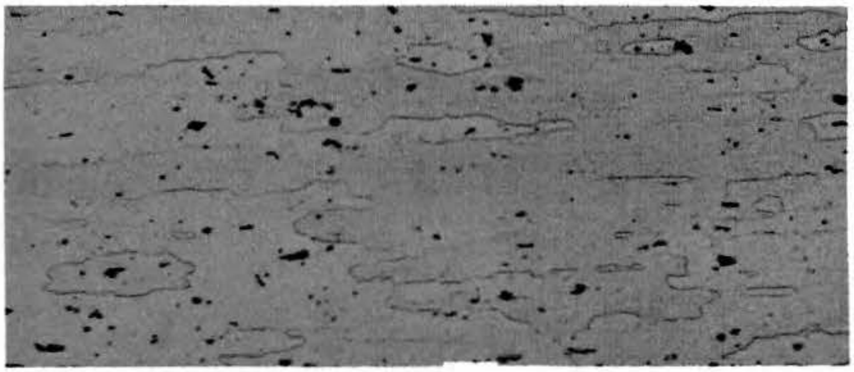
Specimen A1
40% Reduction
MAG. 250X

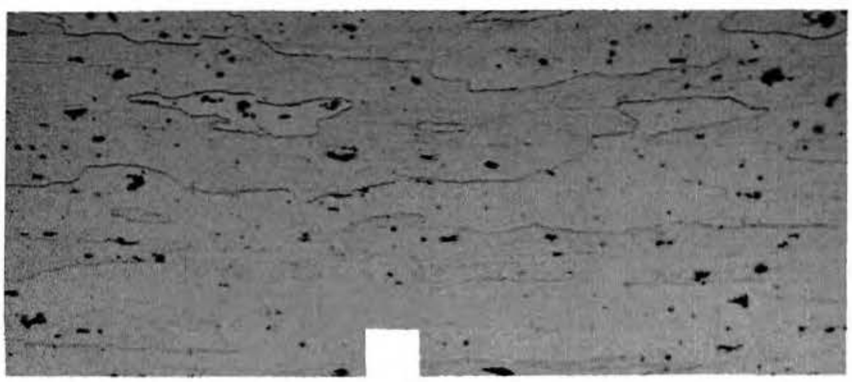
FIGURE 48 – Microstructural Comparison of 0.390 inch Thick Material Reduced in the Annealed Condition



Specimen A4
Control
MAG. 250X

Specimen W1
10% Reduction
MAG. 250X





Specimen W3
25% Reduction
MAG. 250X

Specimen W4
40% Reduction
MAG. 250X

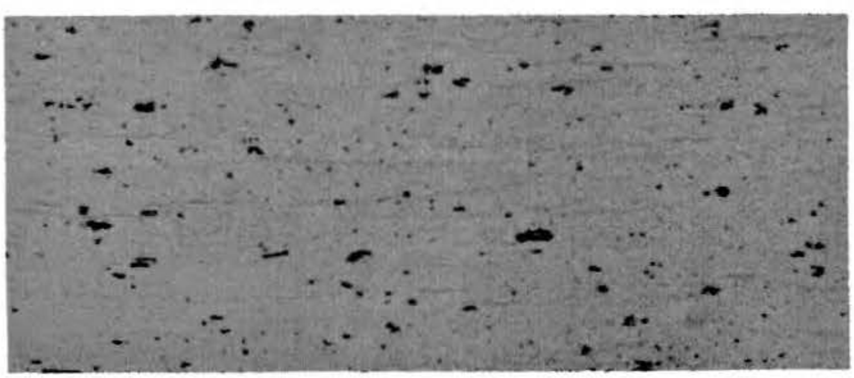
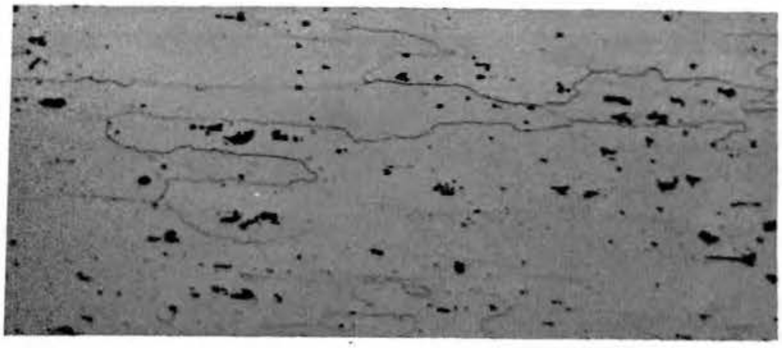


FIGURE 49 – Microstructural Comparison of 0.390 Inch Thick Material Reduced in the Solution Heat Treated Condition

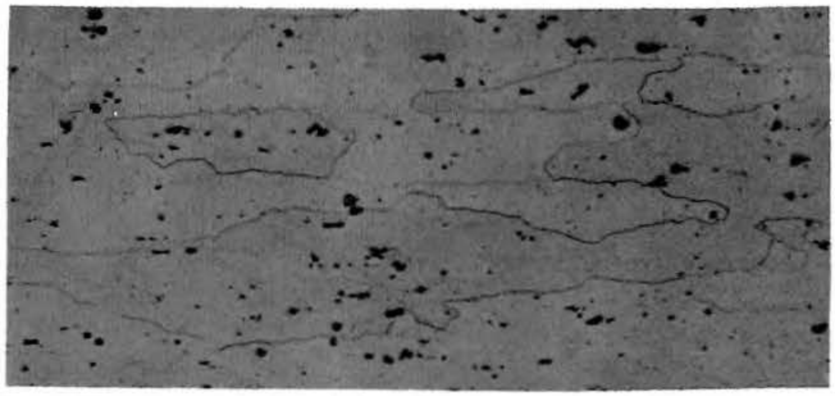
·osion was greatly improved. The microstructures are shown in ıre 50.

The 0.250 inch thick material reduced in the solution heat treated iition and only aged after reduction exhibited very directional, ıly worked grains which demonstrated progressively less resistance exfoliation corrosion attack. These microstructures are shown in ure 51.



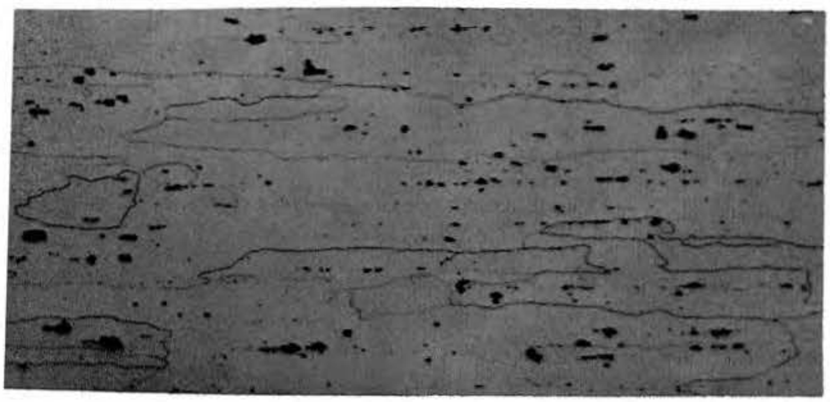
Specimen W4X
Control

MAG. 250X



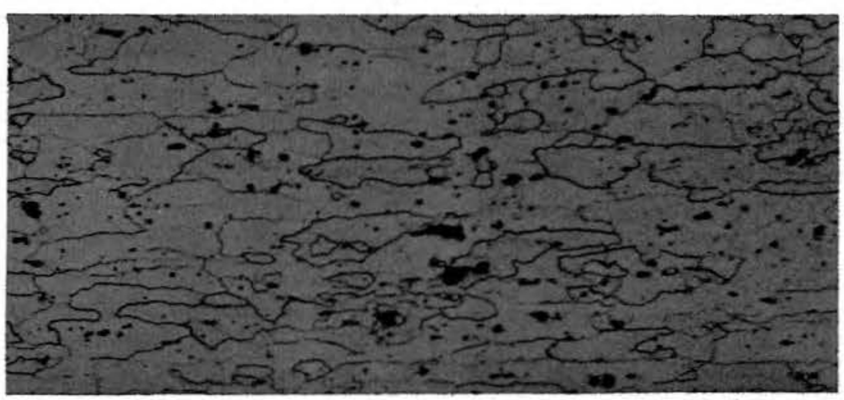
Specimen A4X
10% Reduction

MAG. 250X



Specimen A3X
25% Reduction

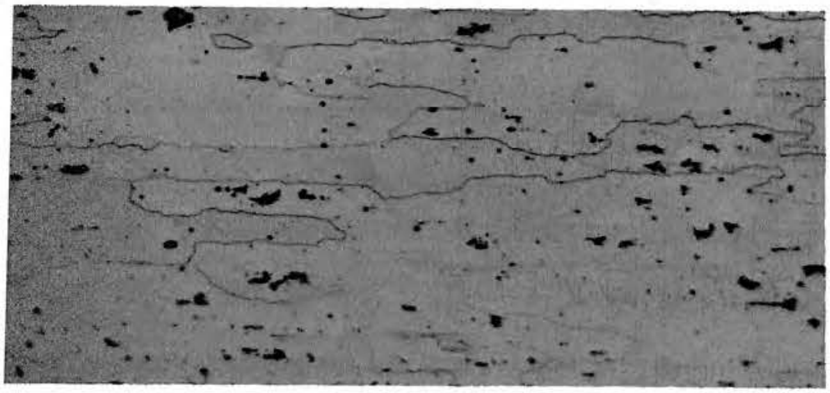
MAG. 250X



Specimen A2X
40% Reduction

MAG. 250X

FIGURE 50 – Microstructural Comparison of 0.250 Inch Thick Material Reduced in the Annealed Condition

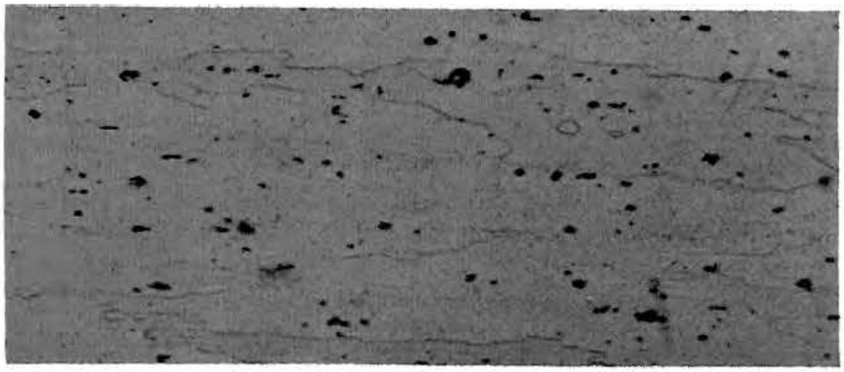


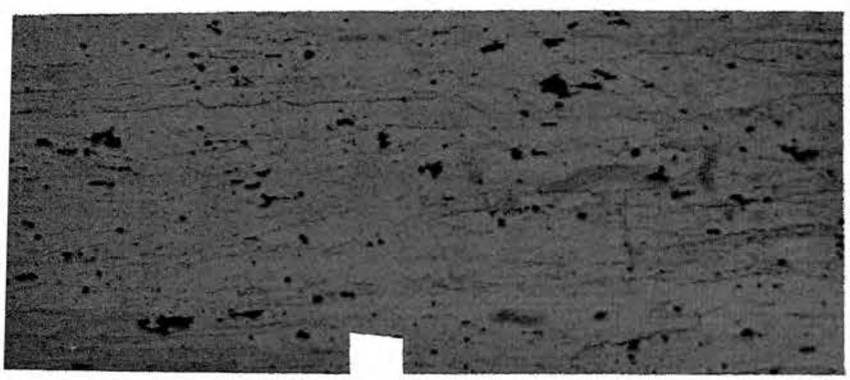
Specimen W4X
Control

MAG. 250X

Specimen W2X
10% Reduction

MAG. 250X





Specimen W3X
25% Reduction

MAG. 250X

Specimen W1X
40% Reduction

MAG. 250X

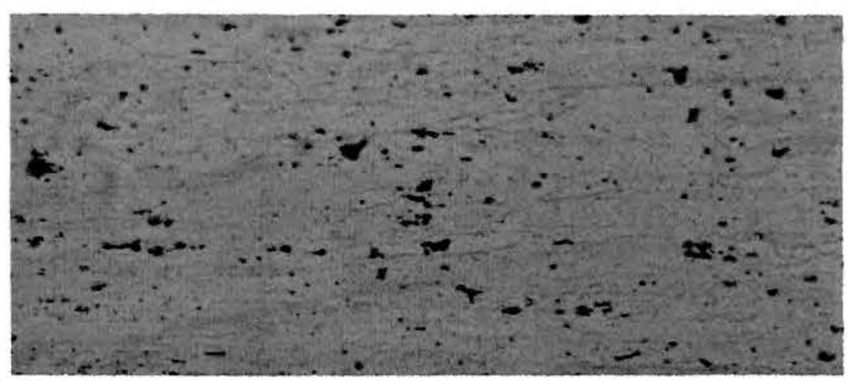


FIGURE 51 – Microsturctural Comparison of 0.250 Inch Thick Material Reduced in the Solution Heat Treated Condition

IV. CONCLUSIONS

The Effect of Thermal-Mechanical Processing on the Strength of 7178 Aluminum Alloy

It has been demonstrated that by altering the aging schedule for erial deformed in the solution heat treated condition that a high ength condition can be maintained. Deformation in this evaluation sisted of reducing the material 10%, 25%, or 40% and then aging at conventional aging temperature of 250 $\pm$ 5°F for shorter aging es. The strength of the T6 condition was successfully maintained the material aged to the T8 condition, and the cold reduction did impair the ductility of the material.

The difference in Rockwell "B" hardness between the "as-quenched" dition and before aging, for the material reduced in the annealed dition, is probably a result of GP zone formation due to $2\frac{1}{2}$ days room temperature aging. The hardness increase for the material luced in the solution heat treated condition is a result of both cold work and GP zone formation.

The more rapid increase in electrical conductivity as artificial ing begins, for the material reduced in the solution heat treated ndition, results from the accelerated aging due to the induced cold rk in this material.

The Effect of Thermal-Mechanical Processing on the Exfoliation Corrosion Resistance of 7178 Aluminum Alloy

The primary objective of this evaluation was to improve the foliation corrosion resistance of the 7178 aluminum alloy by ducing additional nucleation sites adjacent to the grain boundaries,

·eby reducing the width of the PFZ. If the width of the PFZ is ıced, and the grain boundaries are jogged, the electrochemical lic path required for exfoliation corrosion to proceed should be :rrupted and make attack more difficult. Since electron micro- ›y was not available for this study, the following conclusions based on the work of other investigators (3, 9, 18, 25) who per- ned electron microscopy on aluminum alloys, cold reduced in the ıtion heat treated condition.

In this evaluation, all the material reduced in the annealed lition exhibited strain free grains after solution heat treatment 370°F, water quenching, and aging at 250°F. In this material, it assumed that the width of the PFZ remained fairly constant and the r variable which would influence exfoliation corrosion was the ln shape and size. For both thicknesses of material, the exfoli- on corrosion resistance improved as the grain size became strain e, smaller and more equiaxed. This is in agreement with the work Lifka and Sprowls (3) which concludes that certain mill products e a grain structure which is optimized for exfoliation corrosion occur. Other mill products, where recrystallization occurred to duce an equiaxed grain structure, had improved exfoliation corro- n resistance.

The material reduced in the solution heat treated condition is umed to have additional nucleation sites induced adjacent to the in boundaries (9, 25) which would allow increased precipitation occur thereby reducing the width of the PFZ. This should, as viously stated, result in an electrochemical anodic path which is difficult for exfoliation or intergranular corrosion to follow. er, exfoliation has also been reported (3) to follow striations n grains. The material, reduced in the solution heat treated tion, exhibited increasingly directional, highly strained grains ie amount of cold reduction was increased. As the directionality imount of work in the grains increased, the material's resistance cfoliation corrosion decreased. It therefore appears that in the series of aluminum alloys, resistance to exfoliation corrosion ore dependent upon grain size and shape, and upon the subgrains, ations, and other strain induced anomalies within the grains than the width of the PFZ, which had been considered the primary electro- ical anodic path along which exfoliation proceeded.

It has been assumed, in the past, that the mechanism which pro- s exfoliation corrosion is similar to the one which causes stress osion cracking in the 7000 series aluminum alloys. That is, the ary corrosion path is along the PFZ. Therefore, reducing the h of the PFZ or equalizing the potential difference between the the grains, and the material adjacent to the grain boundries, s accomplished by the specially developed aging treatment, will ove the material's resistance to stress corrosion cracking and sequently to exfoliation corrosion. However, this work indicates icing the width of the PFZ alone, as was accomplished in the material l reduced in the solution heat treated condition, does not inhibit reduce a material's susceptibility to exfoliation corrosion. ther electrochemical anodic path, just as favorable as the PFZ t be present in the material which was only artifically aged after eduction. Since the exfoliation corrosion resistance decreased percentage of cold reduction of the solution heat treated al increased, favorable corrosion path must have been intro- e.g., subgrains, striations within grains, etc.

hese conclusions are consistant with the fact that the overaging ents developed to improve exfoliation corrosion have only been ally successful. In some 7178 aluminum alloy material these ents are successful in providing improved resistance to exfol- corrosion, while in others the extended aging does not improve loy's exfoliation corrosion resistance. In addition, some 7178 ium alloy material in the high strength, artificially aged, T6 ion has been found to be as resistant to exfoliation corrosion material aged to the special condition.

To determine the exact path along which the exfoliation corrosion ogressing, it is suggested that any future work on this subject le electron microscopy. It is felt that only after this path is ted can effective action be taken to develop treatments which l completely or inhibit exfoliation corrosion.

This work also indicates that a mill reduction schedule could be oped for each thickness of plate material between 0.150 to 0.750 s, the most susceptible thickness range, to induce enough work into aterial to effect recrystallization during the subsequent solution treatment cycle. This technique would result in material which mproved resistance to exfoliation corrosion without incurring trength loss by developing strain free, equiaxed grains during the ion heat treating cycle.

Lastly, this evaluation has demonstrated that the NaCl plus SO_2 s a considerably more severe environment for evaluation of a ial's resistance to exfoliation corrosion than is the acidified spray environment.

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